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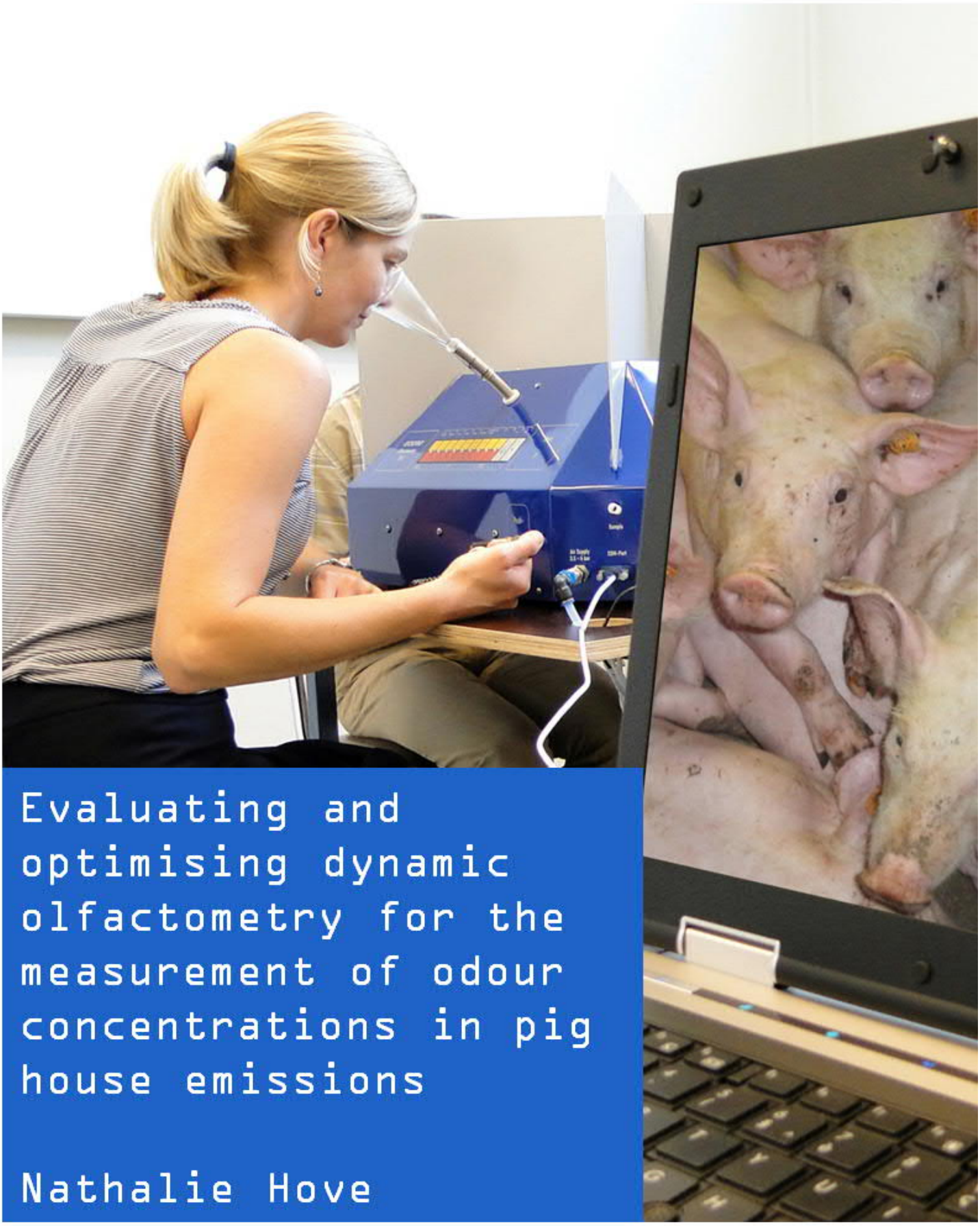

Evaluating and optimising dynamic olfactometry for the measurement of odour concentrations in pig house emissions

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Evaluating and optimising dynamic olfactometry for the measurement of odour concentrations in pig house emissions

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SUMMARY

Pig husbandry is an important sector in food production within Europe. In Belgium, specifically, 1.061 million tonnes of pig meat were produced in 2016, of which 0.702 million tonnes were exported (VLAM, 2016). In that manner pig husbandry contributes largely to the economy in Belgium. Pig husbandry in Belgium, is mainly concentrated in the region Flanders, which holds 5.829 million pigs, covering 94 % of the Belgian pork stack (Statistics Belgium, 2016; VLAM, 2016).

Under the influence of the national and European regulations, more attention is paid to the impact of intensive livestock on the environment. Agricultural activities do not only influence the water and soil system (eutrophication, acidification, etc.), they also influence the air quality (for instance, global warming). During livestock production particulate matter, ammonia, greenhouse gases and multiple other inorganic and organic volatiles are emitted. The emitted volatiles can induce odour perception and can cause odour nuisance.

Since 2009, there is a trend towards expanding pig farms. The intensification of pig husbandry potentially results in a larger odour impact, which in regions with a high population density, such as Flanders (6.509 million inhabitants), can result in more odour nuisance. In Europe, odour is one of the most important sources of complaints (Nicell, 2009)

Within Europe, in this context, dynamic olfactometry, according to the CEN EN 13725 : 2003 – standard (CEN, 2003) and using n-butanol as a reference gas, is set forth as the standard method to measure total odour concentrations and emissions, originating from industrial and agricultural activities. This method is also used to measure the efficiency of odour abatement techniques. Important bottlenecks however appear when practicing this method. Therefore, this standardized measuring technique was critically evaluated within this PhD, with the purpose of measuring odour concentrations present in pig houses. Measures to enhance the precision of the olfactometric method were also investigated.

This thesis handles the following main aspects:

1) Making an inventory of the important critical points when applying dynamic olfactometry according to the CEN EN 13725:2003 standard: focussing on the measurement of odour concentrations and emissions originating from intensive livestock.

2) Determination of the sources of variation during odour measurements.

3) Studying the applicability of the currently used reference gas, n-butanol, for the selection of odour panels for measuring environmental odours such as pig house odour.

4) Formulating measures that can be applied on laboratory level to improve the precision of dynamic olfactometry

To be able to perform this research, within this PhD, an odour laboratory was started up and developed. The requirements of the European standard for olfactometry were thereby followed.

The composition of the odour emissions providing from pig husbandry, as well as the impact of these emissions on the environment are discussed, namely the perception of these odorants' mixtures by neighbours and the origin of odour nuisance were explained in **chapter 1**. The different methods to measure odour and odour nuisance and the assessment framework for odour were presented too. A critical literature study, concerning the bottlenecks in the application of olfactometry according to the CEN (2003) standard was also performed with the purpose of measuring odour emissions originating from intensive livestock. As appears from the envisioned research aspects, the representativeness of the current reference gas and the large measuring uncertainty on olfactometric measurements mainly came forward as important bottlenecks. In **chapter 2**, the research objectives and the structure of this thesis are given.

In **chapter 3**, the development of the olfactometric odour laboratory is discussed. The infrastructural-technical aspects are addressed as well as the selection of the odour panel. In the view of the build-up of the laboratory, it was very important to attain an odour free room and odour free dilution air. The constitution of a qualified odour panel was indispensable. The selection and constitution of an odour panel, large enough to perform regular odour measurements was a major challenge. The first panel selection measurements showed that a small fraction of the tested persons fulfilled both selection criteria, set by the European standard for olfactometry. In total, 80 persons were tested of which 32 were found qualified to participate in odour measurements. The panel

selection measurements were organised periodically, also dependent on the availability of colleagues whom participated voluntary in the odour tests. The results of a first exploratory and intensive odour sampling and measuring campaign in a pig house in Diksmuide are also presented in this chapter. In addition the results of a first participation in inter-laboratory comparison tests with n-butanol and environmental odours are described. From these measurements the need for the further expansion and refinement of the existing odour panel came forth. The first measuring results suggested differences in performance (of panellists) between n-butanol and environmental odours. During the inter-laboratory comparison tests, the odour laboratory did fulfil the accuracy requirements of the CEN (2003) standard: this applied to the measurement of n-butanol samples as well as to the measurement of environmental odours. While the measurements of samples of environmental odours were sufficiently repeatable, the n-butanol measurements during the ICO-tests were not repeatable enough. This prompted us to investigate precision enhancing measures for olfactometric measurements. On the other hand it initiated us to study the predictability and transferability of the current reference gas, n-butanol with the purpose of measuring environmental odours.

Chapter 4 aims at providing a better insight into the sources of variation when performing olfactometric measurements and in the representativeness of the reference gas. Therefore a measuring campaign was set up, in which measurements of duplicate samples of n-butanol and pig house air were repeated by in total 11 to 12 panellists per measuring day. 7 measuring days were organised and 7 sampling days preceded them to sample odour from a pig house. Statistical analysis of the results of the odour measurements indicated that amongst the investigated sources of variation (duplicate samples, different panels, panellists and the individual variability of panellists), the individual variation of panellists was the largest source of variation. This source of variation however had no significant influence on the measuring results. During the study of the representativeness of the reference gas, a weak relation was found between individual n-butanol thresholds and the corresponding individual pig house odour thresholds. The basic principle and starting point of CEN (2003), namely that the odour sensitivity of panellists towards environmental odours can be predicted by their sensitivity towards n-butanol, could therefore not be sustained based on the measuring results. The repeatability within individual panellists as well as between different panellists was better for pig house odour measurements than for n-butanol measurements, but no significant difference was found between both odours based on the performed analyses.

In **chapter 5**, measures are investigated to improve the precision of olfactometric measurements. For that purpose, a tool was developed in Matlab, with which the effect

of different panel sizes, of a higher individual repeatability (performance level) of panellists, of a higher number of rounds, and of different odour types on the precision of panels could be studied. Using this tool, 40500 random combinations (compositions) of panellists and odour thresholds were generated in total, including simulations of 5 different panel sizes (4; 5; 6; 7; 8) and of 2 numbers of dilutions' rounds (2; 3) based on measuring data of 2 odour types (n-butanol; pig odour) and allowing to study the effect of 2 panellists' performance levels (good; best). The tool was set up as such that it could calculate the odour concentrations corresponding with these 40500 simulated odour measurements, according to the rules described in the CEN (2003)-standard, including the retrospective screening procedure. These 40500 odour measurements were simulated, using datasets of earlier performed odour measurements by CEN (2003) – qualified panellists, namely individual odour thresholds measured during panellists' follow-up measurements (with n-butanol) on one hand and on the other hand individual n-butanol and pig house odour thresholds, measured during the measurement campaign, described in chapter 4. In practice, this tool allowed to save up 10125 analyses' hours for this precision investigation. The first statistical analysis showed that the panel size, the number of rounds as well as the panellist's individual repeatability significantly influenced the precision of odour measurements of n-butanol. Through a larger panel size the highest precision could be realised. The second analysis, based on n-butanol and pig house odour data, showed that the odour type, the panel size as well as the number of rounds significantly influenced the precision of panels. Remarkable was the significantly higher precision for pig house odour measurements compared to n-butanol measurements. Former experiments in literature already pointed in that direction. Odour type had the largest influence on the precision of odour measurements, but is a fixed condition. The largest precision could be realized, when using a higher panel size compared to a larger number of rounds, for pig house odour as well as for n-butanol. The repeatability factor between two repetitions of the same odour sample could be improved from 1.90 to 1.54 (crit. CEN < 3) by enlarging the panel size.

In the general discussion (**Chapter 6**) the effect of different precision enhancing measures (more samplings, a higher panel size, a higher number of rounds) on the evaluation of the quality of efficiency measurements of odour reducing techniques was studied. Mainly for measuring techniques with an odour reducing potential of 40 % or lower, the application of the investigated precision enhancing measures appeared to be necessary. In practice, it is important to weigh the measuring costs against the anticipated level of precision. The supplementary costs for a larger panel, longer or more measuring sessions therefore were also calculated. Based on these assessments, it

appeared that applying a larger panel size compared to a smaller panel size and more rounds or more samplings, could in certain cases be economically more advantageous, while it could even lead to more precise measurements. In this chapter, the practical consequences of the use of n-butanol as a reference gas were also addressed, as well as the advantages of using the odour simulation tool. Also olfactometry was situated amongst the different existing odour measurement methods.

Finally, the general conclusions and a number of suggestions for further research were proposed in **chapter 7**.

SAMENVATTING

De varkenshouderij is een belangrijke sector in de voedselproductie binnen Europa. In België werd 1,061 miljoen ton varkensvlees geproduceerd in 2016, waarvan 0,702 miljoen ton geëxporteerd werd (VLAM, 2016). Op die manier draagt de varkenshouderij in belangrijke mate bij tot de economie in België. De varkenshouderij in België is voornamelijk geconcentreerd in de regio Vlaanderen, die met 5,829 miljoen varkens, 94 % van de varkensstapel in België omvat (Statistics Belgium, 2016; VLAM, 2016).

Onder invloed van de nationale en Europese regelgeving, gaat er meer aandacht uit naar de impact van de intensieve landbouw op het leefmilieu. Landbouwactiviteiten beïnvloeden niet alleen het water- en bodemsysteem (eutrofiëring, verzuring, enz.), maar ook de luchtkwaliteit (bv. opwarming van de aarde). Tijdens deze activiteiten komt o.a. fijn stof, ammoniak, broeikasgassen en andere anorganische en organische vluchtige verbindingen vrij. De geëmitteerde vluchtige verbindingen kunnen geurperceptie induceren en geurhinder veroorzaken.

Sinds 2009 is er een trend naar schaalvergroting in de varkenshouderij. Deze intensifiëring van de varkenshouderij brengt potentieel ook een grotere geurimpact met zich mee, wat in dichtbevolkte gebieden zoals Vlaanderen (6,509 miljoen inwoners), tot meer geurhinder kan leiden. In Europa is geur één van de belangrijkste bronnen van klachten (Nicell 2009).

Binnen Europa wordt in die context, dynamische olfactometrie volgens de CEN EN 13725: 2003 – standaard (CEN, 2003) en met n-butanol als referentiegas, vooropgesteld als standaardmethode voor het meten van de totale geurconcentraties en –emissies afkomstig van industriële activiteiten en de landbouw. Ze wordt ook aangewend om de efficiëntie van geurreducerende maatregelen te meten. De praktische uitvoering van deze methode blijkt echter belangrijke knelpunten te vertonen. Deze gestandaardiseerde meettechniek werd daarom kritisch geëvalueerd binnen dit doctoraat, met het oog op het meten van geurconcentraties in varkensstallen. Maatregelen om de precisie van de olfactometrische meetmethode te verhogen werden ook onderzocht.

Dit proefschrift behandelt volgende hoofdaspecten:

1) Inventariseren van de belangrijke knelpunten bij de toepassing van dynamische olfactometrie volgens de CEN EN 13725:2003 standaard: met bijzondere aandacht voor het meten van geurconcentraties en -emissies afkomstig uit de intensieve veehouderij

2) Bepalen van de bronnen van variatie tijdens geurmetingen

3) Onderzoek van de bruikbaarheid van het huidig referentiegas, n-butanol in de selectie van geurpanels met het oog op het meten van omgevingsgeuren zoals geur afkomstig uit varkensstallen

4) Formuleren van maatregelen die op laboratoriumniveau kunnen ingezet worden ter verhoging van de precisie van dynamische olfactometrie

Om dit onderzoek te kunnen uitvoeren, werd binnen dit doctoraat een geurlaboratorium uitgebouwd en opgestart. De voorschriften van de Europese standaard voor olfactometrie werden hierbij gevolgd.

In **hoofdstuk 1** wordt de samenstelling van de geuremissies afkomstig uit de intensieve veehouderij alsook de impact van deze emissies op de leefomgeving uitgelegd, m.n. de waarneming van deze geurmengsels door omwonenden en het ontstaan van geurhinder. De verschillende methoden om geur en geurhinder te meten alsook het beoordelingskader voor geur in Vlaanderen kwamen ook aan bod. Tevens werd een kritische literatuurstudie gevoerd aangaande de knelpunten bij de toepassing van olfactometrie, volgens de CEN (2003)-standaard, voor het meten van geuremissies afkomstig uit de intensieve veehouderij. Zoals uit de vooropgestelde onderzoeksaspecten blijkt, kwamen uit deze studie vooral de representativiteit van het huidig referentiegas n-butanol en de grote meetonzekerheid op olfactometrische metingen als belangrijke knelpunten naar voren. In **hoofdstuk 2** worden de onderzoeksobjectieven en de structuur van de thesis aangegeven.

In **hoofdstuk 3** wordt de uitbouw van het olfactometrisch geurlaboratorium beschreven. Hierbij komen zowel de infrastructurele en technische elementen aan bod alsook de selectie van het geurpanel. Bij de opbouw van het geurlaboratorium was het zeer belangrijk om een geurvrije ruimte en geurvrije verdunningslucht te bekomen. Het samenstellen van een geschikt geurpanel was onontbeerlijk. De selectie en samenstelling van een voldoende groot geurpanel voor regelmatige geurmetingen vormde een grote uitdaging. De eerste panelselectiemetingen toonden dat een beperkt percentage van de geteste personen voldeed aan beide selectiecriteria, die de Europese

norm voor olfactometrie stelt aangaande de kwalificatie voor het geurpanel. In totaal werden 80 personen getest, waarvan 32 geschikt werden bevonden om deel te nemen aan de geurmetingen. De panelselectiemetingen werden periodiek georganiseerd, mede afhankelijk van de beschikbaarheid van collega's die op vrijwillige basis deelnamen aan de geurtesten. Ook de resultaten van een eerste verkennende, intensieve geurstaalname- en meetcampagne in een varkensstal in Diksmuide worden belicht in dit hoofdstuk. Daarnaast worden de resultaten van de eerste deelname aan ringtesten met n-butanol en omgevingsgeuren beschreven. Uit deze metingen bleek de nood aan de verdere uitbreiding en verfijning van het toenmalige geurpanel. De eerste meetresultaten suggereerden verschillen in performantie (van panelleden) tussen n-butanol en omgevingsgeuren. Tijdens de ringtesten voldeed het geurlaboratorium aan de accuraatheidsvereisten van de CEN (2003)-standaard: dit zowel voor het meten van n-butanol stalen als voor het meten van omgevingsgeuren. Terwijl de metingen van stalen van omgevingsgeuren voldoende herhaalbaar waren, waren de n-butanol metingen tijdens de ringtesten onvoldoende herhaalbaar. Dit zette ons aan tot het onderzoeken van precisie-verhogende maatregelen voor olfactometrische metingen. Anderzijds initieerde het ons ook tot een studie van de voorspelbaarheid en transfereerbaarheid van het huidig referentiegas, n-butanol voor metingen van omgevingsgeuren.

Hoofdstuk 4 heeft tot doel een beter inzicht te verschaffen in de bronnen van variatie tijdens olfactometrische metingen en in de representativiteit van het referentiegas. Hiertoe werd een meetcampagne opgezet, waarbinnen de metingen van duplicaatstalen van n-butanol en varkensstallucht herhaald werden door in totaal 11 tot 12 panelleden per meetdag. Er werden 7 meetdagen georganiseerd en daaraan voorafgaand 7 staalnamedagen in een varkensstal. Statistische analyse van de resultaten van de geurmetingen gaf aan dat onder de onderzochte bronnen van variatie (duplicaatstalen, verschillende panels, verschillende panelleden en de individuele variabiliteit van panelleden), de grootste bron van variatie de individuele variabiliteit van de panelleden was en dit voor beide geurtypes. Deze variatiebron had echter geen significante invloed op de meetresultaten. Tijdens de studie van de representativiteit van het referentiegas, werd een zwakke relatie gevonden tussen individuele n-butanol drempels en de overeenkomstige gemeten (individuele) stalgeur drempels. Het basisprincipe en uitgangspunt van CEN (2003), m.n. dat de gevoeligheid van panelleden voor omgevingsgeuren kan voorspeld worden a.d.h.v. hun gevoeligheid voor n-butanol, kon bijgevolg op basis van de meetresultaten niet ondersteund worden. Zowel de herhaalbaarheid binnen individuele panelleden als tussen verschillende panelleden was beter voor metingen van varkensstallucht ten opzichte van deze van n-butanol, maar er

werd geen significant verschil tussen beide geuren gevonden op basis van de uitgevoerde analyses.

In **hoofdstuk 5** worden maatregelen onderzocht ter verhoging van de precisie van olfactometrische metingen. Hiertoe werd een tool ontwikkeld in Matlab, waarmee het effect van verschillende panelgroottes, een hogere individuele herhaalbaarheid (prestatieniveau) van panelleden, een hoger aantal rondes, en van verschillende geurtypes op de precisie van panels kon bestudeerd worden. Met behulp van de tool werden in totaal 40500 random combinaties (samenstellingen) van panelleden en geurdrempels gegenereerd, waarbij panels van 5 verschillende panelgroottes (4, 5, 6, 7, 8) en 2 verdunningsreeksaantallen (2, 3) gesimuleerd werden en dit op basis van meetgegevens voor 2 geurtypes (n-butanol, stallucht) en waarmee ook het effect van 2 panellid-performantie-niveau's (goede, beste) kon onderzocht worden. De tool was zodanig opgesteld dat hij de geurconcentraties overeenkomend met deze 40500 gesimuleerde geurmetingen kon berekenen, volgens de regels beschreven in de CEN (2003)-standaard, waaronder de retrospectieve screening procedure. Deze 40500 geurmetingen werden gesimuleerd, gebruik makend van datasets van eerder uitgevoerde geurmetingen, nl. geurdrempels gemeten tijdens panellid-opvolgingsmetingen (met n-butanol) enerzijds en anderzijds de individuele n-butanol en stalluchtdrempels, opgemeten tijdens de meetcampagne beschreven in hoofdstuk 4. In de praktijk liet deze tool toe om 10125 analyse-uren uit te sparen voor het precisie-onderzoek. Een eerste statistische analyse toonde aan dat zowel de panelgrootte als het aantal rondes, als de individuele herhaalbaarheid van panelleden de precisie van geurmetingen van n-butanol significant beïnvloedden. Door het aanwenden van een grotere panelgrootte kon de grootste precisie gerealiseerd worden. De tweede analyse, gebaseerd op n-butanol en stallucht data, gaf aan dat zowel het geurtype, de panelgrootte als het aantal rondes de precisie van panels significant beïnvloedde. Opvallend was de significant hogere precisie voor stallucht metingen in vergelijking met n-butanol metingen. Geurtype had de grootste invloed op de precisie van geurmetingen, maar is niet te variëren. De grootste precisie kon gerealiseerd worden via een grotere panelgrootte vergeleken met een hoger aantal rondes en dit zowel voor stallucht als voor n-butanol. De herhaalbaarheidsfactor tussen 2 herhalingen van hetzelfde geurstaal kon verbeterd worden van 1.90 naar 1.54 (crit. CEN < 3) door het verhogen van de panelgrootte.

In de algemene discussie (**hoofdstuk 6**) wordt het effect van verschillende precisie-verhogende maatregelen (meer staalnemingen, hogere panelgrootte, hoger aantal rondes) op de evaluatie van de efficiëntie van geurreducerende technieken bestudeerd.

Vooral voor de uitmeting van technieken met een reductiepercentage van 40 % of lager bleek de aanwending van de onderzochte precisie-verhogende maatregelen zeker noodzakelijk. In de praktijk is de afweging tussen de meetkosten en het gewenste niveau van precisie belangrijk. De bijkomende kosten voor een groter geurpanel, langere of meer meetsessies werden daarom ook berekend en voorgesteld. Uit deze berekeningen bleek dat het aanwenden van een grotere panelgrootte in bepaalde gevallen economisch voordeliger kon zijn dan een lagere panelgrootte én meer rondes of meer staalnames, terwijl ze zelfs tot preciezere metingen konden leiden. In dit hoofdstuk worden ook de praktische gevolgen van het gebruik van n-butanol als referentiegas aangekaart, alsook de voordelen van de geursimulatietool en een situering van olfactometrie tegenover verschillende bestaande geurmeetmethoden.

Tot slot worden de algemene conclusies en een aantal suggesties voor verder onderzoek voorgesteld in **hoofdstuk 7**.

TABLE OF CONTENTS

LIST OF ABBREVIATIONS

Abbreviation	Description
β	Regression coefficient
BAT	Best Available Techniques
conc.	Odour concentration
crit.	Criterion
CEN Crit. 1	Panel selection criterion of EN 13725 (CEN, 2003) for the panellists' individual repeatability
CEN Crit. 2	Panel selection criterion of EN 13725 (CEN, 2003) for the panellists' individual sensitivity
C.V.	Coefficient of variation
CVlog	Coefficient of variation calculated on the logarithmic (decimal logarithm) odour concentrations
d	Dilutions' round: a sequence of dilutions of the odour sample
exc.	Exceeding according to CEN (2003)
FEP	Fluorinated ethylene propylene
IPPC	European "Integrated Pollution Prevention and Control" - convention
ITE	Individual threshold estimate
k	Expansion factor
LLA	Lower limit of agreement
Log mean	Logarithm of the geometric mean
LSM	Least square means
MAP	Manure action policy
MD	Mean difference
MKROS	Registration system for environmental complaints
MOPD	Maximum distance of odour perception
N	Total number of …
n	Number
OAV	Odour activity value
OTV	Odour threshold value (ppbv)
ou_E	European odour unit
$ou_E\ m^{-3}$	European odour units per cubic metre
PET	Polyethyleneterephtalate
PM	Particulate matter

Abbreviation	Description
PPL	Panellist's performance level
PTFE	Polytetrafluoroethylene
PTR-MS	Proton-transfer-reaction Mass Spectometry
PVF	Polyvinylfluoride
10^r	Repeatability factor
r	Repeatability limit calculated according to CEN (2003)
r.s.	Retrospective screening according to CEN (2003)
SIFT-MS	Selected ion flow tube Mass Spectometry
$s_{\Delta Z}$	Standard deviation on the ratios between the individual thresholds of panellists and the panel's threshold
$s_{\Delta Z\_env}$	Spread on the ratios between the individual thresholds of panellists and the panel's threshold for environmental odours
$s_{\Delta Z\_but}$	Spread on the ratios between the individual thresholds of panellists and the panel's threshold for n-butanol
SE	Standard error
SLO	Written Living Environment Study
s	Standard deviation
s_r	Repeatability standard deviation calculated according to CEN (2003)
t	Value deduced from student-t-distribution
U	(Expanded) uncertainty
ULA	Upper limit of agreement
Var. Est.	Variance estimate
VOC	Volatile organic compound
x	Individual repeatability according to panel selection criterion 1 in CEN (2003)
y	Individual sensitivity according to panel selection criterion 2 in CEN (2003)
Y_{env}	Pig odour ratio
Y_{nbut}	n-butanol ratio
ΔZ	Ratio between a panellist's individual threshold estimate and the panel's threshold for an odour sample, calculated following CEN (2003)
98-P	98 percentile, indicating that during 98 % of the time the respective immission level is not exceeded in that area

CHAPTER 1

GENERAL INTRODUCTION

CHAPTER 1: GENERAL INTRODUCTION

1.1 Importance of pig farming in Flanders

Intensive livestock faming is an important food production system (Melse et al., 2009a; Melse et al., 2009b; Walgraeve et al., 2015; Hove et al., 2012) in many countries and adds great value to their economies both in terms of export of products and employment (Melse et al., 2009a; Melse et al., 2009b).

Pig production is very important around the globe. China is the main producer of pig meat (46%). The European Union comes on the second place with 20 % of the global pig meat production, while the United States of America account for 10 % of the total pig meat production (Statistical office of the European Union, 2013). Within Europe, Spain grows the largest number of pigs (29.231 million pigs); followed by Germany (27.376 million); France (12.793 million pigs) and Denmark (12.281 million pigs) (Statistical office of the European Union, 2016). The Netherlands (11.881 million pigs) and Belgium (6.176 million pigs) fulfil the fifth and the eight place (Statistical office of the European Union, data 2016). Within Europe, Belgium accounts for 5 % of the total pig meat production, equivalent with 1,061.000 tonnes product weight (Statistical office of the European Union, 2016).

Pig husbandry in Belgium is mainly concentrated in Flanders' region, which holds 5.829 million pigs over 4331 pig farms, covering 94 % of the Belgian pork stack (Statistics Belgium, 2016; VLAM, 2016). Between 2000 and 2015, the number of pig farms decreased strongly in Flanders (with 54 %, going from 8940 to 4145 pig farms) (Driesen et al., 2016). This decrease in the number of farms, went along with a decrease in the total number of pigs, namely from 7.051 million pigs (2000) to 5.981 million pigs (2015). The decrease in the pig population in Flanders was a result of a more stringent environmental policy, aiming at reducing the nitrate concentrations in ground and surface waters and also due to the buy-up arrangement of the government from 2000 to 2004 by which in total 386 500 pigs were taken out of production (Driesen et al., 2016). The introduction of the manure action policy (MAP III) in 2007 however led to new expansion possibilities for farmers in case they processed their own manure and so the total number of pigs increased again from 2009 onwards. This scaling up of the remaining pig farms is visible in the average number of pigs held per farm. At the moment on average

1346 pigs are held per pig farm (Statistics Belgium, 2016), while in 2000 only 789 pigs were held per pig farm.

The province of West-Flanders houses the largest concentration of pigs in Flanders, namely 57 % of the pig population, and is followed by the province of East-Flanders and Antwerp, which are good for 17 % and 16 % of the pig population respectively (Driesen et al., 2016).

In Flanders a distinction is made between class 1 (more than 1000 pig places for pigs older than 10 weeks) and class 2 farms (between 5 and 1000 pig places for pigs older than 10 weeks) depending on the number of pig places and the area in which the farm is located. Figure 1.1 depicts the location and number of industrial pig farms in Flanders (Geopunt Vlaanderen, 2018). These are class 1 farms, which include more than 2000 places for fattening pigs of more than 30 kg or more than 750 places for sows and are subject to European legislation due to a potentially higher environmental impact. Most of them are localised in the Province of West Flanders and the Northern of Antwerp (Fig 1.1).

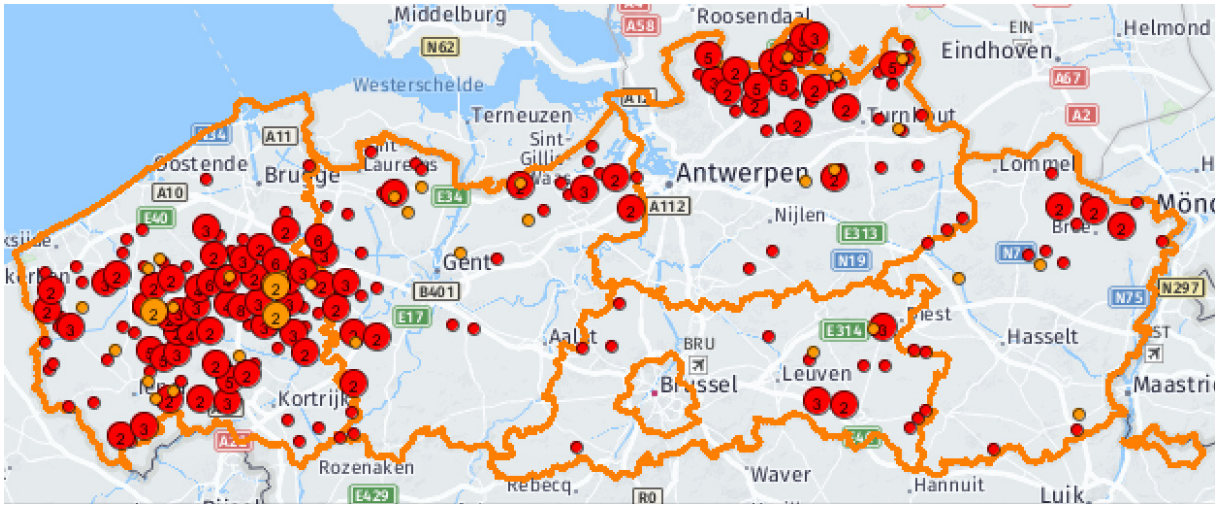


Figure 1.1 Number and location of pig farms in Flanders (in red: fattening pigs, in orange: sows) (Geopunt Vlaanderen, 2018)

Under influence of the European legislation, specifically the IPPC-directive (Integrated Prevention and Pollution Control – directive) (European Commission, 2013), housing systems were developed to reduce the ammonia emission from pig farms (Driesen et al., 2016). Table 1.1 shows the number of pigs per housing system based on an inventory of the VLM of the year 2015 (personal communication department 'Landbouw & Visserij'). As can be seen, only 24 % of the pigs in Flanders are held in low ammonia emission housing systems (Table 1.1). About 48 % of the ammonia emission reduction techniques include end-of-pipe techniques (Table 1.1), whereas 53 % include more source based techniques (e.g.feeding according to needs, frequent removal of manure, optimisation of ventilation rate in the pig house, etc.).

Table 1.1 Number of pigs per housing system and their relative amount in Flanders (VLM, 2015)

Housing system	N of pigs	Relative amount
Traditional housing system	4766687	**76%**
Low emission housing system	1492435	**24%**
	6259122	100%

Low emission housing system	N of pigs	Relative amount
Biobed	17338	1%
Biological scrubber	381228	26%
Chemical scrubber	284313	19%
Air scrubber and slurry	22159	1%
Other	787397	53%
	1492435	100%

1.2 Pig farming as a source of aerial emissions including odour

Pig production has an important influence on the environment and affects the water and soil system (eutrophication, acidification, etc.) as well as the air quality.

Different airborn compounds and pollutants, particulate matter (PM) and gases, are produced inside pig barns (Ulens, 2015). The gaseous pollutants include ammonia (NH_3); greenhouse gases, such as carbon dioxide (CO_2), methane (CH_4), nitrous oxide (N_2O) and numerous other organic and inorganic volatiles. In total, agricultural activities account for 94 % of the ammonia emissions in Flanders (MIRA, 2014). Van Ransbeeck (2013) estimated the yearly emissions of the total pig industry in Flanders to be: 10 771 ton yr^{-1} NH_3, 2.263 million ton yr^{-1} CO_2, 56 036 ton yr^{-1} CH_4, 830 ton yr^{-1} N_2O, 17 ton yr^{-1} PM1, 31 ton yr^{-1} PM2.5 and 398 ton yr^{-1} PM10. Also endotoxins and micro-organisms are present in pig houses (Van Ransbeeck, 2013; Ulens, 2015).

The animals and their waste also produce trace concentrations of volatiles often called “odorants”, because of their effect (Le et al., 2005). A more detailed description of these compounds is given in section 1.4. First a brief description of odour perception will be given.

1.3 Odour perception and nuisance

Odour arises when chemical, volatile compounds are emitted by a source (e.g. decaying organic material, manure applied on the land, …) and dispersed in the air, where they are diluted (meteorologically, topographically) and they can be perceived by the human nose (Van Langenhove et al., 2007). The perception of odour thus is a signal for the presence of chemical compounds in the air. Odour perception occurs when the concentration of an odorant exceeds the detection threshold of the human sense of smell (Driesen et al., 2016). The detection threshold of an odorant is the minimum concentration of that odorant that induces odour perception. The perception of odour is a complex process, involving integration of signals at different levels (Figure 1.2): when air is inhaled, most of the chemical compounds travel directly to the lungs. The odorous compounds that stay behind in the nose can reach the 1 cm² smell epithelium (per nasal cavity), also called the mucosa olfactoria (Schamp & Van Langenhove, 1987) (Figure 1.2 b). Odorants interact with olfactory receptors cells (neurons) in the mucosa olfactoria, that send a signal to the olfactory bulb (i.e. the part of the brain where olfactory signals of individual neurons are converged and processed further). In the olfactory bulb, the signals are firstly converged in the glomeruli and secondly in the mitral cells.

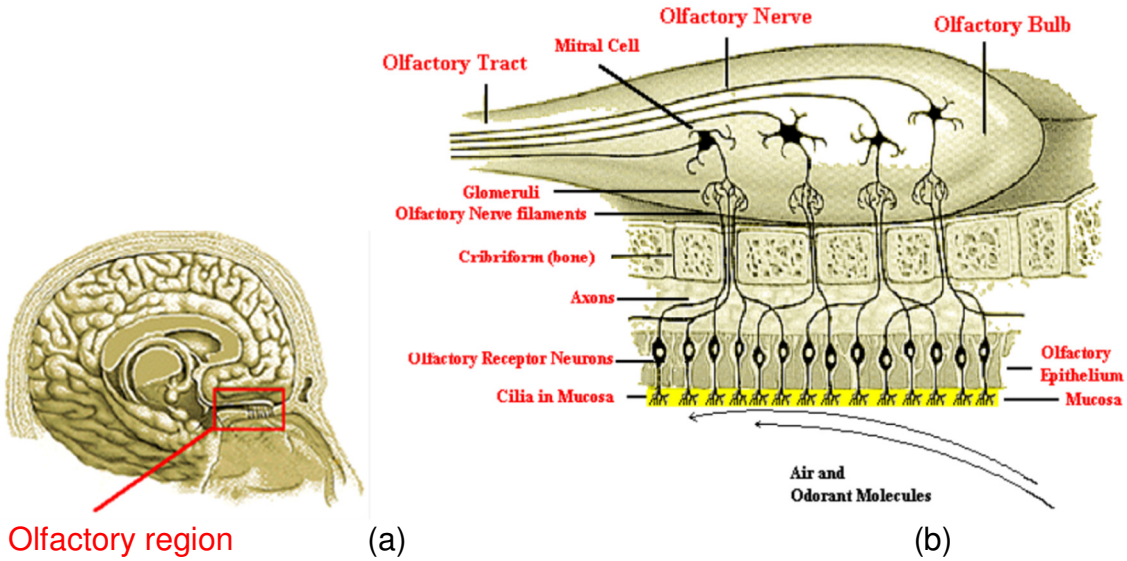


Figure 1.2 Odour perception (Source: Leffingwell Reports, Vol. 2 (No. 1), May, 2002) : (a) Olfactory region, (b) Magnified olfactory region

From the mitral cells on, the signals are sent further in the brain through splitting olfactory nerves, which then connect with the hypothalamus and the corpus amygdaloïdeum in the brain. These centra are involved in the regulation of hunger, social behaviour, pain, etc (Schamp & Van Langenhove, 1987).

A large series of receptor proteins in the mucosa olfactoria, that can interact with different sorts of functional groups, provide that the human nose can register almost an infinite amount of volatile compounds (Schamp & Van Langenhove, 1987). It is known that different odorants activate different combinations of olfactory receptors and result in a unique olfactory code (Malnic et al., 1999; Buck et al., 2004). Slight modifications in the molecular structure of an odorant can generate a different code (Malnic et al., 1999) and can result in a big change in the perceived odour (Malnic et al., 1999; Buck et al., 2004). A mixture of odorants can induce a totally different impression than its individual compounds (Schamp & Van Langenhove, 1987).

The perception of an odour by an individual is influenced by different factors such as the intensity of the odour (strong, weak), the nature of the odour (sour, sweet), the hedonic character of the odour (pleasant, unpleasant) and a few psychological factors such as previous experience and attitude against the odour source (Driesen et al., 2016; Schamp & Van Langenhove, 1987). A distinct and one time perceived odour can at most induce a temporary sense of nuisance for the individual (Schamp & Van Langenhove, 1987). When an unpleasant odour is perceived more frequently and when its intensity increases, odour nuisance (Chapter 1.5) arises (Driesen et al., 2016). **Odour nuisance** therefore is defined as the cumulative result of repetitively perceived disturbances due to odour and that is characterized by an altered behavior (Van Langenhove et al., 2007). In this definition 'cumulative' and 'repetitive' indicate that odour nuisance does not refer to a temporary hindrance, but to a hindrance due to repeated exposure to odorants (Van Langenhove et al., 2007). It manifests itself through a changing behaviour of the perceiver, that can include complaining, closing the windows, remaining out of the garden or by giving negative responses during inquiries or interviews (Van Langenhove & Defoer, 2002). The severity of the nuisance depends on various factors such as the nature of the emitted substances and their quantity as well as on the properties of the emitting source (http://www.compendiumvoordeleefomgeving.nl/indicatoren/nl0291-Geurhinder%3A-bronnen-en-beleid.html?i=13-45). Eventually the response to the presence of odorants in supra-threshold concentrations (concentrations above the detection threshold), can be a collective response, when a certain fraction of the population is hindered and comes forward as a group against the odour problem through actions (Driesen et al., 2016, Schamp & Van Langenhove, 1987). When investigating odour problems, nuisance at group or population level is mostly considered.

1.4 Pig farm odours

The odorants produced in farms (more than 300) constitute a complex and dynamic livestock odour mixture of volatile fatty acids (e.g. acetic acid, propionic acid), ammonia and amines (e.g. methylamine), sulphur compounds (e.g. hydrogen sulphide, dimethyl sulphide) and indoles and phenols (e.g. skatol) (De Bruyn et al., 2001, Yu et al., 2010; Trabue et al., 2011). Most of the odorants are produced by anaerobic microbial conversions of feed (proteins, carbohydrates and lipids) in the gastrointestinal tract of the animals and due to anaerobic decomposition and transformation of livestock wastes, including manure, urine, spilled water, wash water, bedding material, both in the manure pit and on the fouled floor of the animal house (Mackie et al., 1998; Hamon et al., 2012; Van Huffel et al., 2016; Yu et al., 2010).

The odour of the feed and the animals themselves (skin, hair) is mostly not considered offensive, but the odorants generated from manure and manure decomposition are perceived offensive (Mackie et al., 1998). Manure namely is a complex mixture of endogenous secretions, undigested dietary residues, bacterial cells and other metabolic end-products (Mackie et al. 1998). The odorants are emitted into the environment, when manure undergoing degradation has a surface exposed to the atmosphere (Mackie et al., 1998). The produced odorants can adsorb on PM, clothing and building surfaces (Yu et al., 2010). Therefore, odour concentrations inside the animal house constitute an important air quality parameter for both the animals present and for the worker (Yu et al., 2010).

Both the temperature and the moisture content inside the barn affect the rate of microbial decomposition (Yu et al., 2010). When the temperature increases, the pollutants tend to transfer to the gas phase, rather than to stay in the liquid phase (Yu et al., 2010). As follows, the odour production varies seasonally and diurnally with changing outdoor and indoor climatic conditions and changing animal conditions (weight, …) (Yu et al., 2010).

The odorants leave the pig house through ventilation and when the manure is applied on the land. They are diluted further with air in the atmosphere, whereby their concentration decreases. The concentration of the odorants, however, can remain high enough at 100 metres, sometimes even 5 to 10 km from the source to induce odour perception and this due to the high sensitivity of the human sense of smell (Schamp & Van Langenhove, 1987).

In table 1.2, the detection threshold range of some odour compounds, present in pig houses, are given. There is a large spread on the detection thresholds of individual compounds, as reported in literature (Table 1.2) (De Bruyn et al., 2001; Van Huffel et al., 2016). This is due to the different approaches and methods used for determining these detection thresholds. There is no standardized measurement protocol or verification protocol to verify the thresholds of

individual compounds. The detection thresholds of pig house odorants also cover a wide range (Table 1.2), in the sense that they can differ by several orders of magnitude (Van Huffel et al., 2016) (Table 1.2). Even structurally related compounds can show very diffent detection thresholds. As an example, the detection threshold of methyl amine is at a concentration of 2500 µg/m³, while the detection threshold of trimethyl amine is already at a concentration of 0.7 µg/m³. Some of the compounds of livestock odours have a very low detection threshold, which means that a small amount of these compounds already can be detected (Driesen et al., 2016). An example is methane thiol, which is detectable by the human nose at a concentration of only 1 microgram per cubic metre.

The total concentration of the different compounds of livestock odour do not directly reflect their possible odour impact, since the detection threshold can differ largely among different compounds (Van Huffel et al., 2016, Table 1.2). For instance, a compound such as methane thiol can have a larger odour impact in an odour mixture than carboxylic acids, even when present in a lower concentration, because the detection threshold of methane thiol is much lower than that of carboxylic acids (Van Huffel et al., 2016).

Table 1.2 Odour detection threshold range of different pig house odorants, based on the results of different studies (adapted from Van Huffel et al., 2016)

Compound group	Compound name	Odour detection threshold range (µg m^{-3}) *
Volatile fatty acid	**Acetic acid**	10 - 10^6
	n-Valeric acid	10^{-1} - 10^3
Nitrogen	**Ammonia**	10 - 10^5
	Trimethyl amine	10^{-2} - 10
Sulphur	**Hydrogen sulphide**	10^{-1} - 10^3
	Methane thiol	10^{-4} - 10^2
Phenols	**Phenol**	10 - 10^5
Indoles	**3-methyl indol (Skatol)**	10^{-4} - 10^3

Even when the concentrations of the different individual compounds of livestock odours are below the human perception thresholds, the intensity of the total odour mixture can be very strong and its' overall odour well perceivable, through the aggregate effect of the numerous compounds (Yu et al., 2010, Schiffman et al., 2001).

The perception of livestock odorants can differ among humans. In that context, figure 1.3 shows a distribution of individual detection thresholds for hydrogen sulphide based on a test population of 200 persons: a ratio of 5 was found between the P84 (percentile) and P16.

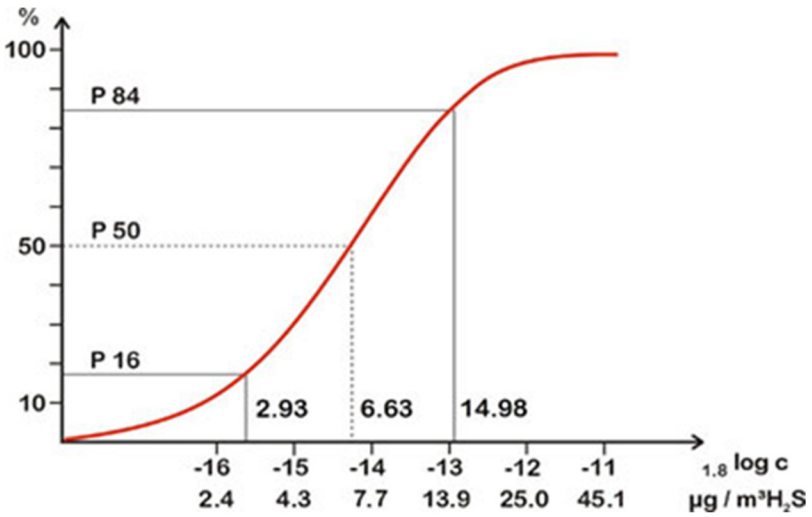


Figure 1.3 Distribution of human olfactory thresholds for hydrogen sulphide (Source: Schamp & Van Langenhove, 1987)

1.5 Odour nuisance related to pig houses

Pig house odorants can have an offensive odour, e.g. the sulphur compounds, the organic acids, indoles, phenols and amines (Table 1.3) (Van Huffel et al., 2012; Heynderickx et al., 2013).

Table 1.3 Odour character of some typical pig house odorants (Le et al. 2006)

Compound group	Compound name	Odorant's smell
Organic acids	Acetic acid	Pungent, vinegar
	Propionic acid	Fecal
	n-Valeric acid	Fecal
Amines	Ammonia	Sharp, pungent, irritating
	Methyl amine	Putrid
	Trimethyl amine	Ammonia-like
Sulphur	Hydrogen sulphide	Rotten eggs
	Dimethyl sulphide	Rotten, Stench
	Methane thiol	Rotten, Garlic, Putrid
Phenols	p-cresol	Fecal
Indoles	3-methyl indol (Skatol)	Fecal, Nauseating

Exposure to the pig house odorants can induce severe odour nuisance, especially for people living in the vicinity of these farms (Kai & Schäfer, 2004; Hansen et al., 2012; Trabue et al., 2011; Walgraeve et al., 2015; Kai & Schäfer, 2004; Hove et al., 2016; Nimmermark, 2011). Especially, when high densities of pig house operations coincide with high population densities, such as in Flanders (6.509 million inhabitants versus 5.829 million of pigs), severe odour nuisance can arise.

Although in most cases there is no direct relation between the odour of substances and their toxicity, odour can give rise to acute physical symptoms, such as nose and eye irritation and respiratory difficulties (Schinasi et al., 2011) and it can more generally affect human health and the quality of life in rural communities (Cole et al., 2000; Thu, 2002; Bullers, 2005; Blanes-Vidal et al., 2009a; Blanes-Vidal et al., 2009b; Mirabelli et al., 2006; Wing et al., 2008; Nimmermark, 2004; Radon et al., 2004; Donham, 2010). Next to that, Eyckmans et al. (2011) found that the value of houses can devaluate with 4 % in regions with moderately to severe odour nuisance. Nuisance problems related to livestock (in general) occur more often nowadays, since livestock facilities have become more concentrated and have increased in size (Donham et al., 2007; Blanes-Vidal et al., 2009; Melse et al., 2009a; Melse et al., 2009b). In Flanders, pigs farms have expanded since 2009 on as a result of the manure action policy (Section 1.1). This intensification of livestock farming occurred while more people moved from the cities to

traditional rural areas (Walgraeve et al., 2015; Libby & Sharp, 2003; Blanes-Vidal et al., 2009a; Melse et al., 2009b; Gutierrez et al., 2014; Melse et al., 2009a; Pillai et al., 2010), not always being ready to accept the perception of livestock odours (Hogberg et al., 2005; Blanes-Vidal et al., 2009a).

As a consequence, offensive odours are an important cause of public complaints to regulatory agencies in Europe and North America (Leonardos, 1995; Nicell, 2009). In Flanders specifically, the SLO (the Written Living Environment Study) is a survey that is held every 5 year to assess environmental nuisance (due to noise, odour and light) amongst the Flemish population. The last survey was held in 2013, evaluating the nuisance experienced by a representative sample of the population in the year 2012. Based on this survey, it appreared that 4 % of the respondents were severely to extremely hindered by odour in their environment and 13 % of the respondents (1 on 7 persons) were moderately hindered by odours in their environment (Departement Leefmilieu, Natuur en Energie, 2013). The next survey will be held in the period February-May 2018 and the results will be known by October 2018 (personal communication, department Environment, Flemish Government). Table 1.4 contains the results of the previous surveys in terms of nuisance due to odour (Departement Leefmilieu, Natuur en Energie, 2013). It could be deduced that 1 on 5 persons encountered moderate to extreme odour nuisance in the year 2000, in the years 2003 and 2007 that were 1 on 6 persons and in the year 2012, 1 on 7 persons (Department Leefmilieu, Natuur en Energie, 2013). The goal for 2020 is that only 4 % of respondents would be seriously to extremely hindered by odour and only 12 % would encounter moderate, severe to extreme odour nuisance, or 1 on 8 persons versus 1 on 7 in 2013 (Departement Leefmilieu, Natuur en Energie, 2013).

Table 1.4 Number of respondents hindered by odour according to consecutive SLO-surveys

Year of SLO	**2001**	**2004**	**2008**	**2013**
Number of valid respondents	3082	4884	5096	5287
Amount of severely to extremely hindered respondents (%)	7%	5%	6%	4%
Amount of moderately to extremely hindered respondents (%)	19% 1 on 5	15% 1 on 6	15% 1 on 6	13% 1 on 7

When looking specifically at nuisance due to agricultural odours, it appeared that 73 % of respondents did not encounter odour nuisance from agricultural odours, 21 % was hindered a little by agricultural odours, 4 % of respondents were moderately hindered and 2 % of the respondents encountered severe to extreme nuisance due to agricultural odours in the year 2012 (Departement Leefmilieu, Natuur en Energie, 2013). Moderate to extreme odour nuisance by agricultural odours occured mainly through spreading of manure on the land (4,7% or N = 257 persons hindered), while odour emissions from pig housed accounted for 1,6 % of

the moderately to severely hindered (N = 96 persons) (Department Leefmilieu, Natuur en Energie, 2013). Poultry and cattle farms hindered 0,8% (N = 43 persons) and resp. 0,6 % of the respondents (N = 34 persons) moderately to extremely. Herds for horticulture caused moderate to extreme odour nuisance for 0,3 % of the respondents (Departement Leefmilieu, Natuur en Energie, 2013).

When comparing agriculture with other sources of odour nuisance in Flanders (Figure 1.4), it appeared that agriculture is the 3rd important source of moderate to extreme odour nuisance, after activities of neighbours and traffic (Departement Leefmilieu, Natuur en Energie, 2013). The amount of moderately to extremely hindered (6% or N = 325 persons in 2012) by agriculture odours did not diminish since the beginning of the measurements in 2000 (Departement Leefmilieu, Natuur en Energie, 2013).

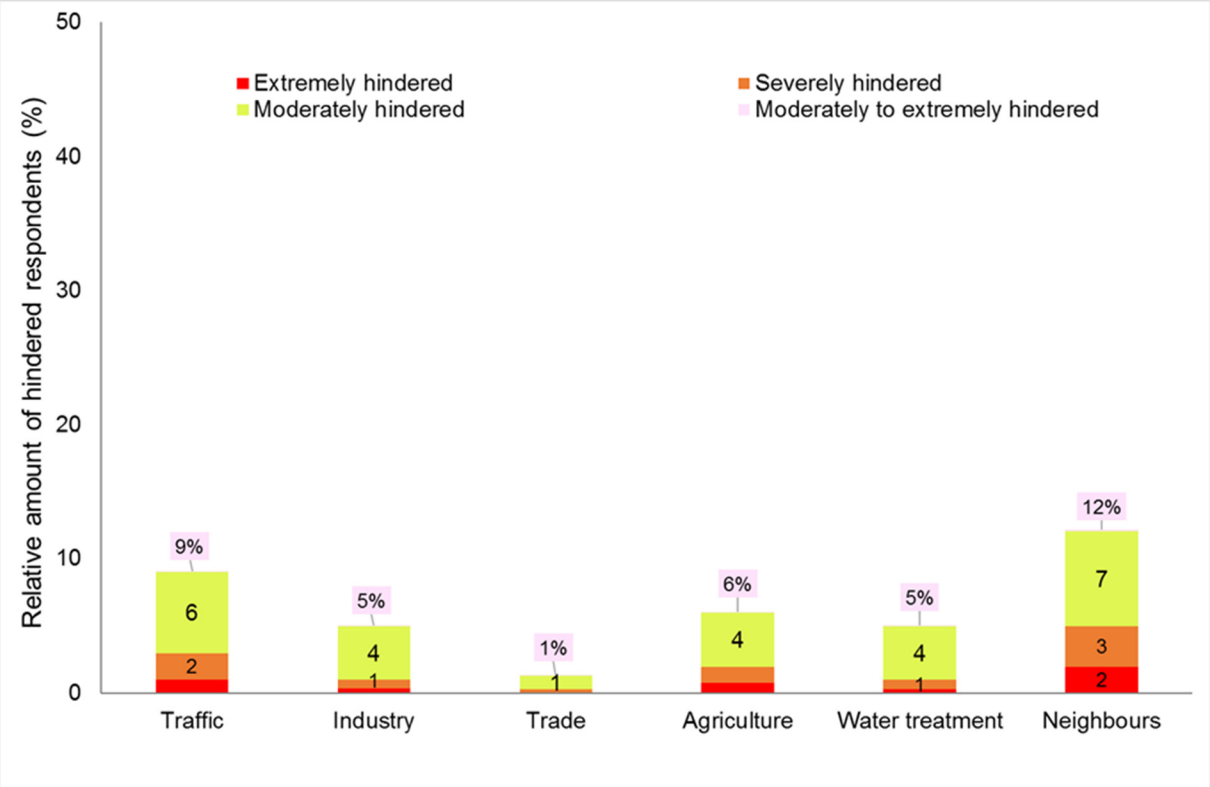
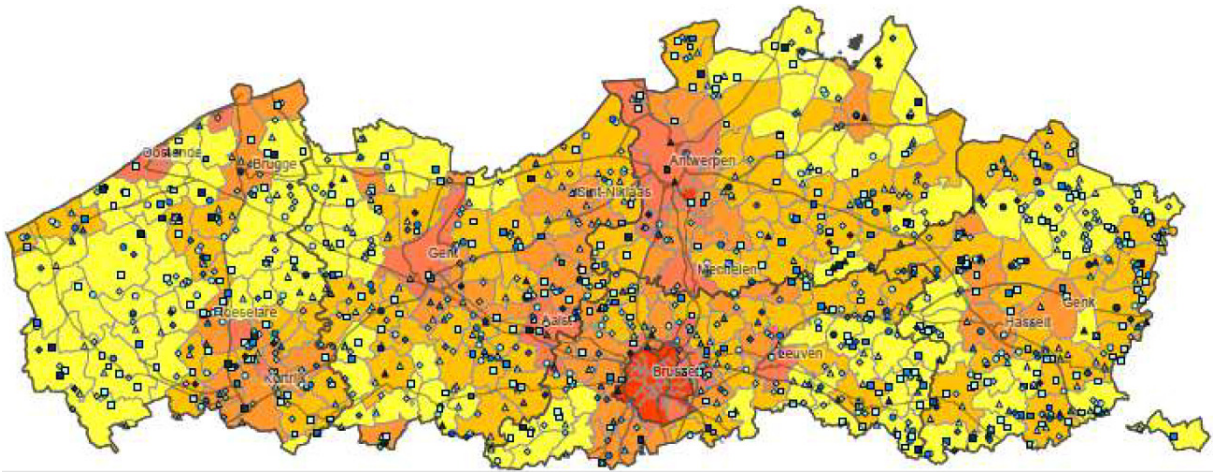


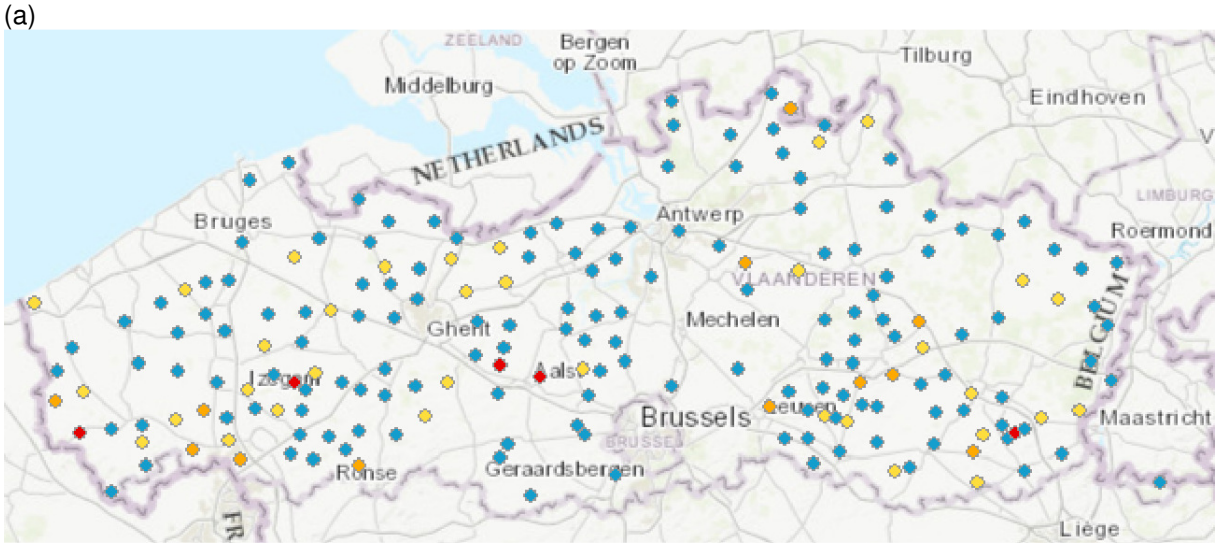
Figure 1.4 Percentage of moderately to extremely hindered by different odour sources (Departement Leefmilieu, Natuur en Energie, 2013)

The SLO also showed a significant difference in the number of respondents that are moderately to severely hindered by odour from agri- and horticulture (Department Leefmilieu, Natuur en Energie, 2013). Only 4% of the respondents encountered moderate to severe odour nuisance in the Province of Antwerp, while in the other Flemish Provinces 6 up to 8 % of respondents were moderately to extremely hindered (Departement Leefmilieu, Natuur en Energie, 2013). The SLO also indicated that the moderately to extremely hindered by agri- and

horticulture are found very dispersed over Flanders (Figure 1.5 a) (Departement Leefmilieu, Natuur en Energie, 2013). This applied as well for those who were a little to severely hindered by odour emissions from pig farms (Figure 1.5 b). Figure 1.5 b was made in the program ArcGis, based on an inventory from the Department Environment from the Flemish Government (personal communication of data applying to pig houses from the SLO of 2013).



(a)



(b)

Figure 1.5: (a) Geographical occurence of the moderately to extremely hindered by odour from agri- and horticulture in Flanders from the years 1999, 2003, 2007 and 2012 (Departement Leefmilieu, Natuur en Energie, 2013)
(b) Geographical occurence of the persons whom where a little (blue), moderately (yellow), severely (orange) and extremely (red) hindered by odour emissions from pig houses in Flanders in 2012 based on 5104 respondents

Another possible indicator for odour nuisance in Flanders are the reports of the Environmental Inspectorate. The Environmental Inspectorate handles complaints concerning class 1 operations as well as complaints about class 2 and class 3 operations that cause serious nuisance. They can be caused by industrial or agricultural sources.

Table 1.5 presents the number of complaints about odour nuisance presented to the Environmental Inspectorate in the past years compared to the total number of complaints presented regarding environmental nuisance (considering odour, sound, etc.). From the table can be deduced that about 30 to 40 % of complaints regarding environmental nuisance were are about odour in the years 2012 till 2016.

Table 1.5 Evolution in the number of complaints due to odour nuisance reported to the Environmental Inspectorate

Environmental Enforcement Report	2012	2013	2014	2015	2016
Number of complaints about odour nuisance	1151	1143	1325	1028	1285
Total number of complaints environmental nuisance	2737	3235	3489	2996	3374
Relative amounts of complaints on odour (%)	42	35	38	34	38

The environmental complaints - registration and follow-up system, MKROS, is a tool, which is used by municipal, provincial and regional gouvernemental bodies that deal with environmental complaints in which data concerning an environmental complaint can be record and monitored in a structured way (https://www.lne.be). It is used for handling complaints from class 2 and 3 operations. In the period 2006 – 2010, about 38000 notifications of environmental problems were done, concerning class 2 and 3 institutions (Van Broeck, 2011). From these notifications, about 23% concerned the compartment air, 21 % concerned sound complaints, etc. (Van Broeck, 2011).

69 % (or N = 6013) of the notifications on environmental problems (N = 8696) in air concerned odour nuisance (the other environmental complaints were about air quality disruption caused by soot, dust, smoke). 6 % (N = 372) of these notifications about odour nuisance were related to agriculture and horticulture (Van Broeck, 2011). Within the target group ‘Agriculture and horticulture’, pig stalls (22% of the complaints or N = 81 reports), the spreading of animal manure on the land (20% or N = 76 reports) and poultry houses (11% or N = 40 reports) were the most important sources of odour nuisance (Van Broeck, 2011). Table 1.6 displays the evolution in the number of odour nuisance reports regarding pig farms from the years 2006 to 2010 (Van Broeck, 2011). The number reporting in the MKROS-system concerning pig farms rose from 7 till 24 from the year 2007 till 2010 (Van Broeck, 2011).

Table 1.6 Evolution in the number of complaints due to pig farms from 2006 to 2010 reported in the MKROS-system (Van Broeck, 2011)

MKROS	2006	2007	2008	2009	2010
Number of odour nuisance complaints related to pig farms	16	7	14	20	24

1.6 Odour measuring methods

One single odour measurement method that allows to describe all atributes (or even detection only) of an odour perception does not exist. The relation between the dose of the odour emitted by the odour source, including the chemical composition of the odorants and their individual concentrations, and the corresponding response (from odour perception to odour nuisance) of neighbouring residents is so complex, that odour problems are analysed by performing measurements at different levels of the disturbance chain.

Firstly, the source strength of the odour source can be determined by measuring the odour concentration and emission through air samples taken at the odour source and olfactometric measurements in the laboratory.

1.6.1 Dynamic olfactometry

Olfactometry is a sensory measuring method, which uses the human nose as a sensor. Air samples of 10-50 L are taken at the emission source in sampling bags, which then, within 30 hours after sampling are presented through a dilution device (the olfactometer) to a panel of qualified assessors (Figure 1.6). Only qualified assessors or panellists are allowed to participate in the olfactometric analyzes, as prescribed by the European standard for olfactometry (CEN, 2003). The qualification is based on individual perfomances with regard to the reference substance, n-butanol (Chapter 3.1.2).


(a)

(b)

Figure 1.6 (a) Odour sampling (b) Olfactometric measurement in the odour laboratory

Odour sampling (Figure 1.6 a) is performed following the 'lung principle' (CEN, 2003): the sampling bag is placed in a vacuum vessel and is connected to the air to be sampled via a

sampling tube. A vaccum pump is connected to the outside of the vessel and sucks air out of the vessel, creating an underpressure in the vessel, so that the sampling bag can be filled through the sampling tube with air of its' environment.

In the laboratory, the odorous sample is presented in various dilutions to a panel of qualified assessors (the panellists) through sniffing ports and using a calibrated olfactometer (air-dilution device) (Figure 1.6 b).

The olfactometer dilutes the odorous air sample stepwise with odourless air in predefined ratios. In figure 1.7, an example of the olfactometer output of one series of dilution steps, presented to 4 assessors (HN, MEC, HL and ABH) is given. The dilution steps are altered with reference air (this is air that is treated in such a way that it is technically as odourless as possible) and blanks (i.e. intentionally presented odourless air). The blanks are indicated by a '0' in the olfactometer output (Figure 1.7). The reference air is not indicated in the olfactometer output, but in practice always follows a presented dilution or blank. When a panellist detects an odour in a presentation, he/she must indicate this by pressing the 'Yes' button (Figure 1.6 b). When they press the button, a 'Yes' displays in the olfactometer output (Figure 1.7).

Steps	HN	MEC	HL	ABH
0				
0				
16384				
8192				
0				
4096			Yes	
0				
2048	Yes	Yes	Yes	Yes
1024	Yes	Yes	Yes	Yes

Figure 1.7 Example of olfactometer output of one round (one dilutions' series presented to 4 panellists).
The highest dilution at which the panellists perceived the odour of the sample is indicated in green.

The odour concentration of the sample is then calculated based on the panellists evaluations of the presented dilutions of the sample. According to CEN (2003) at least two series of dilutions need to be presented to at least 4 panellists, generating at least 8 individual odour threshold estimates (ITE). An ITE is the estimated detection threshold of a person based on one series of dilutions (CEN, 2003). An ITE of a panellist is calculated as the geometric mean of the highest dilution step at which the panellist can just detect the odour of the diluted sample (distinguish it from odourless air) and the lowest dilution step at which he/she could not yet detect the odour of the sample or distinguish it from odourless air. As an example, the ITE of the panellist HN is 2896, which is the geometric mean of 2048 (highest dilution step

corresponding with detection 'Yes') and 4096 (lowest dilution step and still no detection) (Figure 1.7).

A series of dilutions presented to a panel of assessors is also called a round (CEN, 2003) (Figure 1.7). A minimum of 2 rounds are required according to CEN (2003). An ascending order of stimuli (the odour concentration of the presented dilutions of the sample go up: higher dilution steps are presented before lower dilution steps) (Figure 1.7) or a random order of stimuli can be presented to the assessors within a round.

Olfactometric analysis allows to determine the odour concentration of gaseous sample, expressed in European odour units present in 1 m^3 of gas at standard conditions (ou_E/m^3) (CEN, 2003).
The European odour unit (ou_E) is that amount of odorants that, when evaporated in 1m^3 of neutral gas at standard conditions, produces the same physiological response of a panel (i.e. a detection threshold) as that of 1 European Reference Odor Mass (EROM), evaporated in 1 m^3 of neutral gas at standard conditions (CEN, 2003). 1 EROM corresponds to 123 µg n-butanol (CAS No. 71-36-3). If 1 EROM is evaporated in 1m^3 neutral gas, this results in a concentration of 0.040 µmol/mol (or 40 ppbv) n-butanol (CEN, 2003).
The numerical value of the odour concentration of an air sample expressed in ou_E/m^3 is the number of dilutions required to reach the detection threshold of the odour panel.
The detection threshold of the panel is assessed through the presentation of minimum 2 rounds of dilutions of the panellists specifically (see above, figure 1.7). The numerical value of the odour concentration of the sample is then calculated as the geometric average of the ITE of the participating panellists during a measurement and after applying the retrospective screening procedure of CEN (2003) on the participating panellists' ITE. The retrospective screening procedure is described in detail in section 5.2.2 and an illustration of the calculations is added in appendix A. Generally, retrospective screening is a procedure included in the CEN (2003)-standard, which aims at detecting exceptional deviant responses of qualified panellists during a measurement, which could be caused by physiological factors (e.g. temporarily decreased or increased odour sensitivity induced by nasal congestion or irritation) or by psychological factors, such as decreased attention.

In analogy, with expressing the sound pressure level in decibels, odour concentrations can also be expressed in the odour-related decibel unit dB_{od} , by multiplying the decimal logarithm (log_{10}) of the odour concentration expressed in ou_E/m^3 by 10 (CEN (2003)). This is due to the logarithmic relation between an odour stimulus and its' perceived intensity, as described by the Weber-Fechner law in CEN (2003).

The odour emission is the amount of odour that is emitted per unit of time from a specific surface and is calculated as the product of the odour concentration, the discharge rate and the discharge surface. The discharge speed can be determined using an anemometer. The odour emission of a source is also the product of the volume flow rate of the source in m^3/h and the odour concentration expressed in ou_E/m^3. The odour emission, calculated on the basis of olfactometric measurements, is expressed in ou_E/s.

Secondly, the odour emission of the source and the odour immissions in the vicinity of the odour source can be determined by sniffing measurements in the field and using dispersion models.

1.6.2 Sniffing team measurements: the plume method

In contrary to olfactometric measurements, sniffing measurements take place in the field. They allow to determine the odour impact of a source on the environment. The sniffing team method is incorporated at Flemish level in a code of good practice (Bilsen et al., 2008).

During sniffing team measurements the area in the field in which the odour of a company can be perceived is being determined by qualified assessors (Bilsen et al., 2008). The assessors must also meet the criteria set for the panellists of olfactometric measurements (Chapter 3.1.2).

To deviate the odour contour, the assessors will traverse the odour plume along the leeward side of the odor source in zig-zag movement until they can no longer detect odour (Fig 1.8) (Bilsen et al., 2008). The track followed is drawn on a map (Bilsen et al., 2008). The assessor indicates on the map the places where he/she detects the odour and where he/she does not perceive the odour (Bilsen et al., 2008). Using these indications the assessor can draw the best fitting odour contour, after traversing the environment of the odour source (Bilsen et al., 2008). He/she can also deviate the maximum distance of odour perception (MOPD) along an imaginary central axis (Bilsen et al., 2008). This is the maximum distance, downwind from the source, where the odour of the source can just be observed, during a certain meteorological state (Bilsen et al., 2008).

At the beginning and at the end of the sniffing measurement the date, hour, cloud level and wind direction are written down by the assessor (Bilsen et al., 2008). The meteorological situation (wind direction, wind force and cloud level) from the nearest synoptic station of the RMI (KMI) is also retrieved after the sniffing measurement (Bilsen et al., 2008).

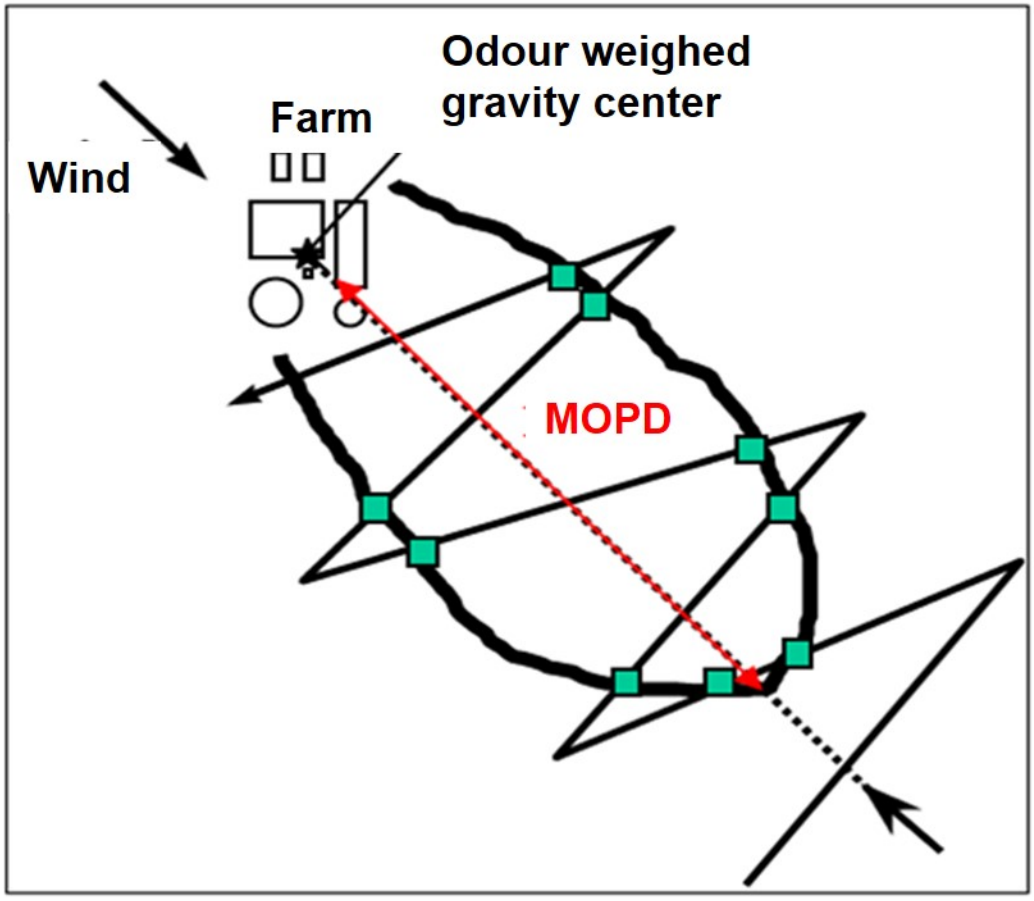


Figure 1.8 Determination of the odour contour and the maximum odour perception distance during sniffing measurements (Source: VLM/AMINAL/TWOL2001/mjp2000-21/88)

Based on the field observations, the maximum odour detection (perception) distance, the source height and the meteorological conditions (wind speed, stability), the odour source strength or the odour emission from the source can be deviated in sniffing units per second (Bilsen et al., 2008). This is done using a short-term dispersion model (su/s) (Bilsen et al., 2008). One sniffing unit per m^3 corresponds to the odour concentration in the field where the odour of the source can just be detected by the observers (Bilsen et al., 2008). This correponds with the odour concentration at the edge of the odour plume or also at the maximum odour detection distance (Bilsen et al., 2008).

There is no clear relation between odour units, determined by olfactometry and sniffing units, determined based on sniffing team measurements (LNE, 2012). One sniffing unit, in practice, is usually more than one odour unit (LNE, 2012).

Once the source strength or the odour emission of the source is determined, one can use dispersion models to calculate the odour concentrations in ambient air on an annual basis (Bilsen et al., 2008).

To get an idea of the impact of the odour source, the immission concentrations (odour concentrations at ground level) in the farm's environment can be expressed in percentiles (Bilsen et al., 2008). A percentile represents a certain percentage of the time that a certain hourly average concentration is not exceeded (Bilsen et al., 2008). Usually 98 percentiles (98-P) are represented. A 98-P of 1su m^{-3} indicates during 98 percent of the time the considered average immission concentration of 1su m^{-3} is not exceeded in the indicated area (Figure 1.9). 98-P also comes down to an exceedance of the respective immission level for 175 hours on yearly basis, in other words one week of exceedance (LNE, 2017) (Figure 1.9).

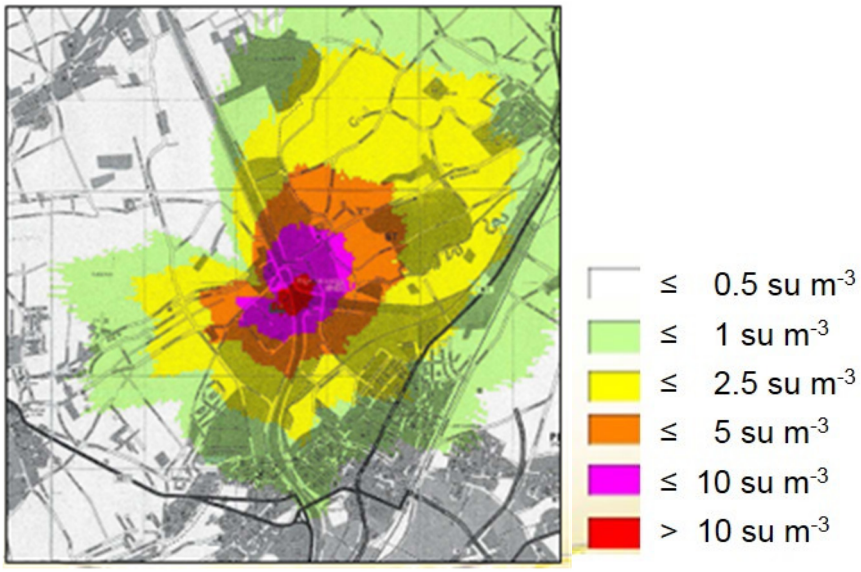


Figure 1.9 98 percentile immission-concentrations determined using sniffing measurements (Source: Raf De Fré en Ilse Bilsen, 2005, Cursus Geurhinder TI-KVIV: Geurmetingen met behulp van snuffelploegen)

The sniffing team method is recently standardized on the European level (EN16841).

Thirdly, the odour components in an air sample taken at the source can be identified and quantified by chemical analysis.

1.6.3 Chemical analysis

Chemical analysis methods (e.g. gas chromatography coupled with mass spectrometry) are performed to identify and quantify the odour compounds in an air sample. Sampling of the odour components is done by enrichment on a sorbent, which is subsequently thermally desorbed and enriched in a cold trap. The odour components are separated from each other by a gas chromatographic column (GC). Quantification of the odour components is done using a detector. All separated compounds are identified by means of a mass spectrometer (MS). The result of a GC-MS analysis of air is usually an extensive list of chemical compounds. Based on literature data (detection thresholds), one can select those compounds that are

responsible for the odour of the source. SIFT-MS (Selected ion flow tube Mass Spectometry) (Van Huffel et al., 2012) and PTR-MS (Proton-transfer-reaction Mass Spectometry) (Van Huffel et al., 2016) are newer techniques. Analysis with those techniques is faster. They do not require sample preparation. Direct sampling in the air stream of the odour source is possible.

Fourthly, sociological methods can be used to evaluate nuisance in a representative sample of the population.

1.6.4 Sociological methods

Sociological measurement methods are applied to verify the nuisance in the population and this on the basis of surveys (LNE, 2010; Van Broeck & Van Langenhove, 2000). Surveys themselves can be performed via face-to-face interviews, in writing (the written life-environmental surveys) or by telephone (for the telephone life-environment survey). Questionnaires with multiple choice questions and/or open questions could be presented to the local residents already long before the survey to investigate the nuisance. The questions concern the perceptibility, the nuisance, odour types and frequency.

The percentage of people affected by odour is determined during the surveys. Surveys are suitable for estimating the severity of an odour problem.

1.6.5 Summary of the methods

In summary, table 1.7 gives an overview of different measurement methods used in odour assessments along with the information they reveal.

Table 1.7 Overview of the methods used for measuring odour and odour nuisance

Measuring methods	Type	Result
Chemical analysis	GC-MS [1]	Identification and quantification of the odorants (ppm or mg/m³)
	PTR-MS [2]	
	SIFT-MS [3]	
Sensory analysis	Olfactometry [4]	Odour concentration [ou_E/m^3]
	Sniffing team method [5]	Odour concentration [se/m³]
Sociological method	Spontaneous complaints [6]	Estimation of the perception of the odour and of nuisance effects
	Enquiries [7]	

[1] Van Huffel et al. (2012)
[2] Van Huffel et al. (2016)
[3] Van Huffel et al. (2012); Heynderickx et al. (2012)
[4] CEN EN 13725: 2003
[5] Bilsen et al. (2008); EN 16841
[6] LNE (2010)
[7] Van Broeck & Van Langenhove (2000)

1.6.6 Linking odour and odour nuisance: a complex dose-response relation

The link between odour emissions from livestock and odour nuisance due to livestock is an example of a complicated dose-response relation.

Dose, within this context, is the occurrence with a certain frequency and periodicity of odour components from livestock in suprathreshold concentrations (i.e. in concentrations, which can be observed by man). (Schamp & Van Langenhove, 1987). A gradation exists in the response, since an increasing dose of odour compounds in the air firstly induces odour perception, when the dose continues to rise the individual sense of nuisance and then the collective sense of nuisance follow (Schamp & Van Langenhove, 1987).

The relation between a change in the odour emission and the corresponding response however is still unknown and very complex, because it is influenced by different fysiological, psychological, psychofysical and sociological factors, that it is difficult to determine the corresponding response. Once the response increases from and individual perception towards the collective sense of nuisance, more factors that are difficult to control and quantify play an important role (Schamp & Van Langenhove, 1987). Therefore with the measurement of the dose, it is still difficult to predict the response.

1.6.7 Comparison of the measurement methods

In table 1.8 an overview is given of the advantages and disadvantages of each of the measurement methods based on literature. The main points will be discussed here. Compared to other odour impact assessment methods, olfactometry has the advantage that it directly uses the human nose as a sensor and therefore includes the perception effect, following the definition of odour (Chapter 1.3). The second advantage is that odorants present in concentrations below the analytical detection limit of chemical methods will be measured also, since the human nose is a very sensitive odour detector, next to a broad-range odour detector (Brattoli et al., 2011), which can perceive odorants of a wide variety at very low concentrations. Chemical analysis (Van Huffel et al., 2012; Bruneel et al., 2016; Hansen et al., 2014) has the advantage that it allows to measure small changes in concentrations of individual odorants and this with a high accuracy and precision (Gostelow et al., 2001; Munoz et al., 2010).
The sniffing team method (Bilsen et al., 2008; EN 16841) has the advantage that it includes the human nose (qualified assessors) as a sensor and that the odour is directly measured in the field after being naturally diluted by its existing environment (instead of mechanically diluted by the olfactometer) and thus directly measures the odour perceived by neighbouring residents. The advantage of this method is that its' working principle is more closely related to

the functioning of the human sense of smell, namely that the human nose is more adapted to detect changes in odour concentrations rather than statically determine the absence or presence of an odour (working principle of dynamic olfactometry). The European standard for the sniffing team method (EN 16841) has been ratified, unfolding two methods for the direct assessment of odour in ambient air (Van Elst & Delva, 2016; www.olores.org, 2015a). A recent study (Driesen et al., 2016) promotes the use of the sniffing team method to allow the individual evaluation of the odour dispersion of a farm. The disadvantage is that those measurements can only be performed under certain meteorological conditions and are difficult to plan. The model used to calculate the source strength of the odour source (in su/m³) also brings along some uncertainty since discrete classes are used to describe mixing in the air (Van Langenhove & Van Broeck, 2001).
Sociological methods (Driesen et al., 2016; Van Broeck & Van Langenhove, 2000) on their turn have the advantage to directly monitor the nuisance effects in the surrounding of a pig farm, but the results are more subjective and difficult to verify.

In fact, integrating all odour measurement techniques (olfactometry, sniffing team method, chemical and sociological analysis), which could be considered as being complementary (Munoz et al., 2010), does result in the most information concerning an odour situation. Together they can inform on the odour mixture that is formed in the pig house as well as provide an understanding on the dilution of the odour in the environment. The odour concentration that attends the neighbouring houses could be assessed and the effect the odour emission has (nuisance or not) on the neighbours. Together they can also inform on the effectiveness of odour abatement techniques.

1.6.8 Focussing on dynamic olfactometry

In this thesis, the focus will be on dynamic olfactometry, in order to measure the perception by humans, following the definition of odour (Chapter 1.3) in an objective manner (with qualified assessors) and this by measuring at the source of the odour (taking samples within pig houses).

Table 1.8 Overview of the advantages and disadvantages of the different measurement methods

Measuring technique	Advantage	Disadvantage
Olfactometry	Based on odour detection: includes the human nose	Cannot be used to identify the odour source
	Human nose is very sensitive and a broad range odour detector	No information on the composition of the odour mixture
	Contribution of different point sources to the total source emission	Labour intensive: measurements require a qualified odour panel
	can be determined bij taking samples at the different emission points	of min. 4 persons
	Odour concentration can be measured at its source	Difficult to apply for diffuse odour sources and surface sources
	Method is standardized in Europe (CEN EN 13725)	Cannot be used to assess ambient air samples
	Efficiency of odour abatement techniques can be assessed	with a concentration of 5-10 ou_E/m^3
	Output can be used to predict the odour impact of a source,	since the method's limit is about 20-50 ou_E/m^3 (3)
	when applying a dispersion model	The odour from the original source is measured in an artificial
	Determination of odour concentration, intensity, hedonic character	environment (laboratory)
	Range of concentrations: odorants from ppbv up to several thousands	Less precise than other techniques: a factor of 3 is allowed according
	of ppmv (4)	to EN 13725 between replicate measurements of the same sample
		Difficult to evaluate the efficiency of abatement techniques with
		lower reduction levels
Chemical analysis	Odorants can be measured at the source	Does not assess the human perception of the odour
	Composition of complex odour mixtures can be determined	Does not directly inform on possible odour nuisance.
	The identification and quantification of odour compounds allows	Preconcentration of samples can introduce uncertainty.
	to identify the potential odour source and gives information on	Labour intensive
	the odour producing processes	Synergetic, antagonistic and additive effects of odorants
	Specific indicative odorants can be measured	cannot be measured.
	Useful for the design of abatement techniques	
	Can inform on the effect of odour abatement techniques on	
	specific compounds	
	Precise: repeatability GC-MS: 10 % in ppbV range;	
	20-30 % in pptV range (1)	
	Detection limit: 0,2 - 5 µg/m³ (2)	
	Range of concentrations: odorants from 1 ppbv up to 10 ppmv (4)	
	Accuracy: 1 ppbv (4)	

Measuring technique	Advantage	Disadvantage
Sniffing team method	Field measurement	Labour intensive: requires a panel of assessors near the odour source
	Measures the global impact of the odour source in its real (direct) environment	Cannot be used to evaluate the effect of an abatement technique
		Can only be performed at specific weather (atmospheric) conditions
	The effect of diffuse compound sources will be included in the global impact in its' environment	Discrete stability classes insert uncertainty on results of modelling
		Does not give information on individual sources from one plant
	European standardization: EN 16841	
	Can be used to assess the odour at concentrations around the nuisance level	
	Includes the human perception of the odour	
	Farm specific approach is possible	
	Output can be used to predict the odour impact of a source, when applying a dispersion model	
Sociological method	Can Inform on odour nuisance	Influenced by subjectivity of respondents
	Informs on the odour in the environment after emission	Includes the human perception of the odour
		Time consuming, requires specific interview training
		Cannot be used to predict the odour impact of a source
		Does not quantify the odour emission of the source
		Difficult to interprete and verify the results
		Results are influenced by the number of respondents
References:	(1) Dewulf and Van Langenhove 2002	
	(2) Defoer and Van Langenhove 2002	
	(3) Gostelow et al 2000	
	(4) Hamon et al 2012	
	Driesen et al 2016	
	Hayes et al 2014	
	Munoz et al 2010	

1.7 Odour impact assessment and regulations in Flanders

In order to prevent and to control odour nuisance, odour regulations are applied. In Belgium environmental issues are regional legal rights (Driesen et al., 2016) and odour problems are locally addressed.

The legislation to control odour nuisance in Flanders is spread over different laws, decrees, regulations, namely: 'The environmental permits decree of June 28, 1985; 'The Flemish regulation about the environmental permit of February 6, 1991' (Vlarem I), 'The conclusion of the Flemish government adhering to general and sectoral provisions about environmental hygiene of June 1, 1995' (Vlarem II), 'title XVI of the Decree General Provisions concerning the environmental policy (the environmental maintenance decree)' and 'The manure decree'.

The Flemish odour policy is based on the following key-ideas (LNE, 2012):

a) Zero – emissions are not realistic

b) If no odour nuisance occurs, no actions or measures are required

c) If there is odour nuisance, the application of techniques to reduce odour emissions is required

d) Severe odour nuisance is never permissible

In summary, acceptable odour nuisance is strived for in the Flemish odour policy (LNE, 2012). A target value is set, called the zero effect level, which is the concentration level that is achieved downwind and at infinite distance from the emission source. This is the quality level that should be achieved and maintained as long as possible (LNE, 2012). Also a limit value is defined. This is the odour concentration level where severe odour nuisance and structural complaints are expected (Willems et al., 2016). This limit value cannot be exceeded, except in case of circumstances beyond one's control. Acceptable odour nuisance is then the pursuid odour impact level situated in between the zero effect level and the limit value (Figure 1.10) (LNE, 2012). The target and limit values for livestock farms were derived from the study of Van Broeck & Van Langenhove (2000). The target value is based on the relation between the number of respondents that perceived odour and were hindered by it, and the number of residents that only perceived odour in the performed assessment. The limit value was defined by the relation between the number of respondents that perceived odour and were severely hindered by it, compared to the number of residents that only perceived odour (Willems et al., 2016). In figure 1.11 the target and limit values for stand-alone (isolated) pig farms and farms in a cluster of farms are indicated. They are represented as immission levels (expressed in su/m^3 cfr. 1.6.2) that cannot be exceeded in 98 % of the time.

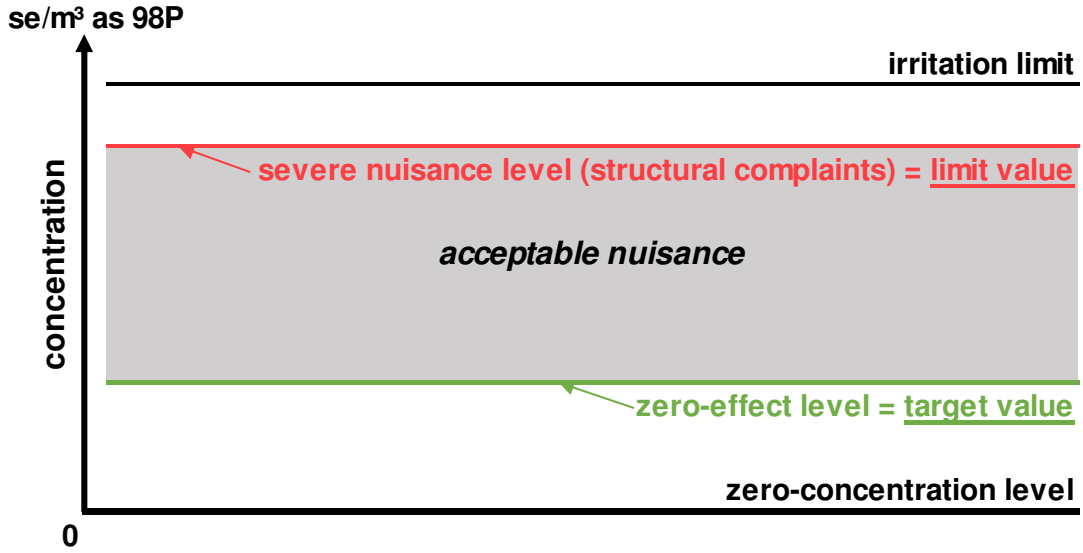


Figure 1.10 Key concepts of the Flemish odour policy (Figure adapted from LNE, 2012)

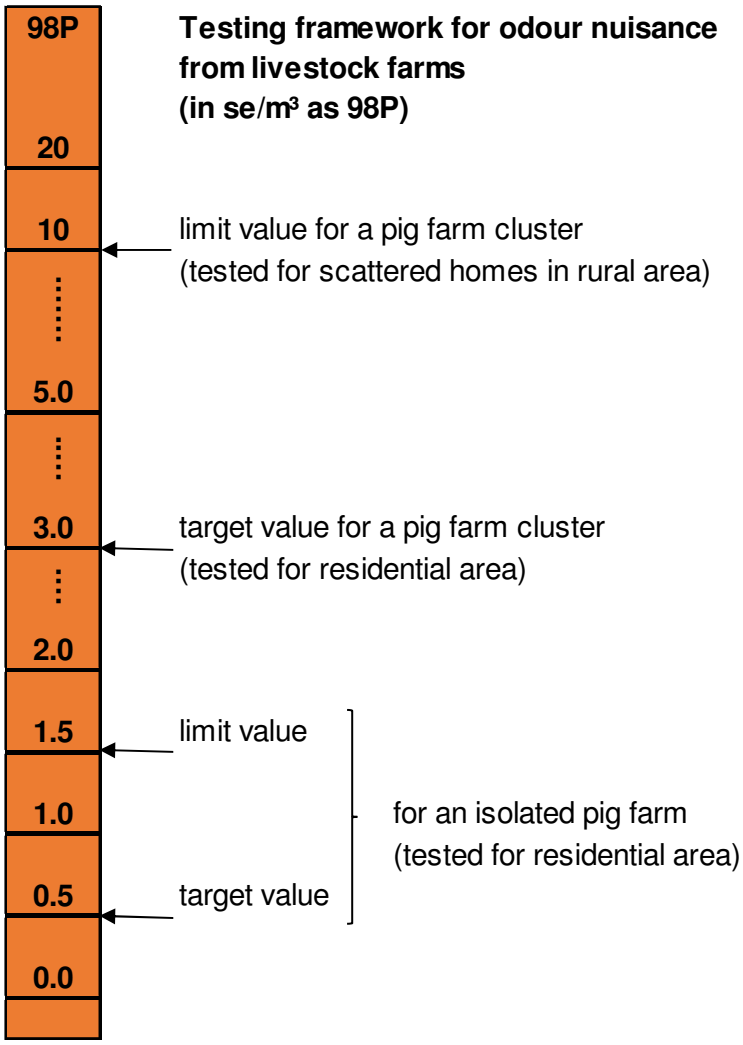


Figure 1.11 Testing framework for odour nuisance from livestock facilities (adapted from LNE 2013)

When evaluating a permit request of class 1 pig houses, an environmental impact assessment is required. In that view, the immission concentrations in the surrounding of the pig farm are predicted. In Flanders, the webtool IMPACT (Immission Prognosis Air Concentration Tool, LNE 2017) is used to model such immission concentrations. This new tool was introduced on January 31st 2017 and is an improved version of the previous IFDM - PC tool introduced by VITO in 1996. The model enclosed in the IMPACT-tool simulates the dispersion of pollutants (also odour) via a Gaussian plume and uses specific hourly averaged meteo – data (LNE, 2017). The odour source needs to be specified in the tool (e.g. source strength, location, number of animals) and also the area of receptors needs to be indicated (LNE, 2017). The source strength of the emission source can be estimated by actual measurements, which have the advantage that project specific data can be used (LNE, 2012). Dynamic olfactometry or the sniffing team method (Chapter 1.6) are then used to estimate the odour source strength, expressed in $ou_E\ s^{-1}$ or $su\ s^{-1}$ respectively. Alternatively, established emission factors can be used in the model as determined by Van Langenhove & Defoer (2002) for conventional pig houses in Flanders by dynamic olfactometry. These emission factors are expressed in European odour units per animal and per second, considering the odour concentration, the ventilation rate and the number of animals present in the pig house. They are represented in Table 1.9.

Table 1.9 Odour emission factors used in Flanders (Van Langenhove & Defoer, 2002)

	Odour emission factors (ou_E animal^{-1} s^{-1})	
Animal type	*Traditional housing*	*Low ammonia emission*
Fattening pigs	29.2	22.7
Weaners	12.1	8.4
Farrowing sows including piglets	84.4	84.4
Mating and gestating sows	57.0	57.0

The emission factors for low ammonia emission housing systems with fattening pigs and weaners (Table 1.9) are derived from the emission factors determined for the corresponding traditional housing systems, by applying the same odour reduction factors as used in the Netherlands (22.2 % and 30.7 % resp.) (Brusselman & Demeyer, 2014; Driesen et al. 2016).

The IMPACT-model calculates the immission concentrations at height of odour-sensitive houses around the farm (the receptor zone) (LNE, 2017). The model visualises the predicted immissions by iso-concentration-contours. The iso-concentration-contours connect places where the same averaged immission concentration is not exceeded for

a certain period of time, indicated as percentiles. As explained in section 1.6.2, usually 98-P are applied. It should be noted that other countries may use a different dispersion model than the Gaussian plume model. Altering the ground model or any of the fixed parameters can result in different predicted immissions. Different results, for instance, could be obtained by applying the V- stacks model, used in the Netherlands (Driesen et al., 2016).

As can be observed from figure 1.11, the impact of certain immission levels is estimated differently as a function of the odour sensitivity of the receptors (Willems et al., 2016). There is a differentiation between:

- Highly odour-sensitive areas, e.g. residential area, schools, hospitals, malls, camping spots, …
- Moderate odour-sensitive areas, e.g. residential area with rural character (if verification against own farm odours)
- Low odour-sensitive areas, e.g. residential area with agricultural character

As a consequence, the standards for isolated farms are more severe than for farms that are part of a cluster of farms (Willems et al., 2016) (Fig. 1.4). The standards for isolated (stand-alone) farms are as follows (Willems et al., 2016):

- Concerning new farms: the target value is 0.5 ou_E/m^3 as 98-percentile and if not achievable using best available techniques, it is set at 1 ou_E/m^3.
- For existing farms the limit value is 1.5 ou_E/m^3 as 98-percentile at the nearest house (if economically possible).

For a farm located in a cluster of other farms, the following standards are set (Willems et al., 2016):

- 3 ou_E/m^3 as 98-percentile as target value, evaluated for highly odour sensitive areas
- 5 ou_E/m^3 as 98-percentile as target value, evaluated for residential area with rural character
- 10 ou_E/m^3 as 98-percentile is the limit value (LNE, 2011) concerning dispersed houses in agricultural areas (houses of neigbouring livestock facilities are not considered here)

The immission standards mentioned in the guideline manual on farm animals (Willems et al., 2016) are expressed in ou_E/m^3 as 98-P, while in the testing framework (LNE, 2012)

they were expressed in su/m³. This although the relation between 1 ou_E and 1 su is not clear yet.

Tables 1.10 and 1.11 illustrate the estimated odour impact of the different immission levels as a function of the odour sensitivity of the considered area. In table 1.10 specifically, the judgement of different immission levels at the nearest house, depending on the odour-sensitivity of the destination is given for an isolated farm. While in table 1.11, the judgement of different immission levels at the nearest house for a farm within a cluster of farms is given.

Table 1.10 Judgement framework for an isolated farm (Willems et al., 2016)

Immissions	Low odour-sensitive destinations *agricultural area*	Moderate odour-sensitive destinations *residential area with rural character*	Highly odour-sensitive destinations *residential area*
> 10 OU_E/m^3 als 98P	Strongly negative effect	Strongly negative effect	Strongly negative effect
3 - 10 OU_E/m^3 als 98P	Moderate negative effect		
1,5 - 3 OU_E/m^3 als 98P	Slightly negative effect	Moderate negative effect	
1 - 1,5 OU_E/m^3 als 98P	Negligible effect	Slightly negative effect	Moderate negative effect
0,5 - 1 OU_E/m^3 als 98P		Negligible effect	Slightly negative effect
< 0,5 OU_E/m^3 als 98P			Negligible effect

Table 1.11 Judgement framework for a livestock farm within a cluster of farms (Willems et al., 2016)

Immissions	Low odour-sensitive destinations *agricultural area*	Moderate odour-sensitive destinations *residential area with rural character*	Highly odour-sensitive destinations *residential area*
> 10 OU_E/m^3 als 98P	Strongly negative effect	Strongly negative effect	Strongly negative effect
5 - 10 OU_E/m^3 als 98P	Moderate negative effect		
3 - 5 OU_E/m^3 als 98P	Slightly negative effect	Moderate negative effect	
< 3 OU_E/m^3 als 98P	Negligible effect	Negligible effect	Negligible effect

According to Vlarem I and II, the owner of a farm needs to take all possible measures to avoid nuisance of any kind (Driesen et al., 2016). They include applying *Best Available Techniques* (BAT) e.g. new pig houses have to be built ammonia emission low. These BAT were introduced under influence of the European Integrated Pollution Prevention and Control convention (European Commission, 2013) for all European pig farms with more than 2000 fattening pig places. The European BREF-document (European Commission, 2003) presents the BAT in a code of good agricultural practices. Amongst these measures different housing systems to control ammonia emissions are described (Chapter 1.1).

In application of the environmental permits decree, the government can even impose specific operational conditions depending on the specific and local circumstances of a farm (Fig 1.12) (Driesen et al., 2016). Mitigating or odour reducing measures can consist of source-oriented techniques (e.g. feed adaptations, ventilation) or end-of-pipe

techniques, cleaning the exhaust air e.g. a biological air scrubber (Ulens, 2015; Vlaamse overheid & Leefmilieu Natuur en Energie, 2012; Van der Heyden et al., 2015).

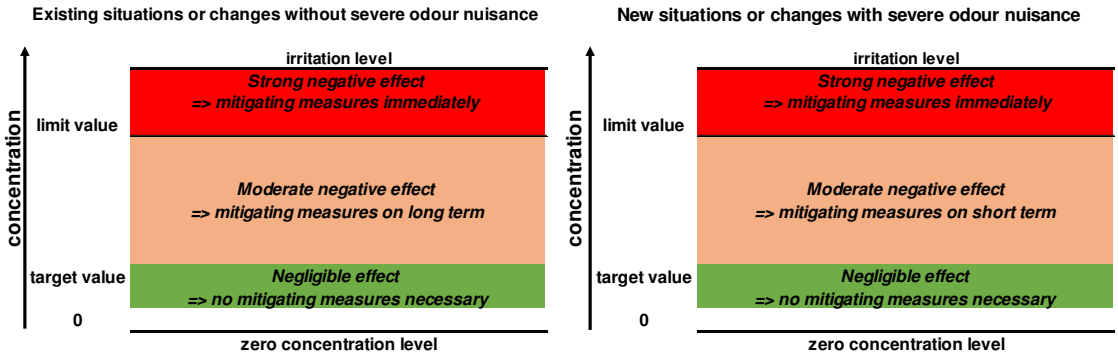


Figure 1.12 Necessity for mitigating measures (LNE, 2012)

When the total assessment shows that there are strong negative effects (the limit value is exceeded), immediate mitigating measures are proposed and the effects of these measures are modeled in the environmental impact report (LNE, 2012). This applies for both re-licenses, changes (changes, expansions or additions) and new projects (Fig 1.12).

In the case of a moderate negative effect, a distinction is made between re-licenses and changes without expecting the existing nuisance to increase, and new installations or re-licenses with changes that are expected to increase the existing nuisance (Fig 1. 12) (LNE, 2012):

- In new situations or changes with an increased impact on nuisance, mitigating measures are needed in the short term and their effects need to be modelled and included in the environmental impact report (including effects being modelled) (Fig 1. 12) (LNE, 2012).

- In the case of existing situations, applying mitigating measures on long-term is required (Fig 1. 12) (LNE, 2012).

When the effects are negligible (Fig 1.5), no mitigating measures should be proposed in the environmental impact report (LNE, 2012).

Specifically, in the environmental impact report, the indicated odour dispersion contours need to include at least (Willems et al., 2016):

- The odour zones: 10-20 ou_E/m^3 ; 3-10 ou_E/m^3 ; 1,5-3 ou_E/m^3 for isolated farms

- The odour zones: 10-20 ou_E/m^3 ; 5-10 ou_E/m^3 ; 3-5 ou_E/m^3 for farm clusters
- The location of the farms from which the emissions are taken into account in the modelling
- The location of the possible influenced houses
- The location of the indicator houses: these are houses that are the most critically located based on the calculated odour concentration and the area where they are located in.

The emission limit of 3 ou_E/m^3 is also presented for the current and the requested situation, taking only into account the emission caused by the individual farm (not by eventual surrounding farms) (Willems et al. 2016). A table needs to be drawn representing the odour immissions at the different indicator houses and including the following information (Willems et al., 2016):

- The individual odour immision in the current permitted situation
- The individual odour immision in the planned situation
- The cumulative odour immision in the current permitted situation (in case of a cluster)
- The cumulative odour immision in the current permitted situation without the individual farm (in case of a cluster)
- The cumulative odour immision in the planned situation

To reduce nuisance effects, Vlarem II also prescribes some distance rules for new facilities and major expansions of pig and chicken farms (Willems et al., 2016). These are certain distances that need to be respected in between the exterior of the farm and the nearest receptor (Willems et al., 2016). The distance rules are a function of the number of animals present and the used housing systems (Willems et al., 2016). The distance rules need to be considered in the odour impact assessment. In case they would be exceeded, mitigating measures also have to be applied (Willems et al., 2016).

The importance of odour impact evaluations is clear from the number of Environmental Impact Reports that are prepared yearly in case of permit requests. In the year 2016, 36% (24 on 66) of the environmental impact reports were prepared with regard to intensive livestock farms (personal communication, departement Omgeving). In the years 2012-2015, 34 % to 43 % of environmental impact reports concerned intensive livestock farms (pig and poultry houses) (personal communication, departement Omgeving).

1.8 Critical literature review on the olfactometric method

In Europe, dynamic olfactometry, performed according to the CEN EN 13725:2003 standard (CEN, 2003), is the most commonly-applied method to assess odour emissions from agricultural sources and from industrial facilities (Klarenbeek et al., 2014; Hayes et al., 2006a; Rzenik et al., 2014;Capelli et al., 2008; Capelli et al., 2012; Capelli et al. 2011). It is an effect-based measurement method, intended to determine the effects of odorants on the perception in humans (Mannebeck, 2017).

As mentioned in chapter 1.7, this measurement method allows to determine odour emission factors (Mielcarek & Rzenik, 2015; Mol & Ogink, 2002), which on their turn are used in atmospheric dispersion models to predict the odour impact zone (iso-concentration contours) of livestock farms and to determine separation distances (Yu et al., 2010; Nicell, 2009; Nimmermark et al., 2005; Nicolas et al., 2008; Romain et al., 2013). Olfactometry can also be used to evaluate the efficiency of odour abatement techniques (Friedrich & Kosmider, 2012; Martens et al., 2001; Melse & Moi, 2004; Miller et al., 2004; Hansen et al., 2014), with the purpose of reducing odour emissions. Olfactometry can also be applied to test agreement with prevailing odour regulations and to verify the estimated odour impact of new or expanded farms (Nicell, 2009; Munoz et al., 2010; McGinley, 2002).

Because of the importance and widespread application of dynamic olfactometry by investigators, for policy makers and developpers of mitigiation techniques, it is vital that this technique meets up to the demands of these applications in terms of performance.

Practitioners of dynamic olfactometry however indicate that the application of the European standard for dynamic olfactometry shows different bottlenecks, which will be discussed here. The performance of dynamic olfactometry, according to CEN (2003), was evaluated by a critical literature review, focussing on the measurement of livestock odours. For this evaluation, critical points at the different stages of the olfactometric CEN-procedure are adressed. Firstly the odour panel selection process is considered, secondly the odour sampling procedure, thirdly the dilution of odour samples in the olfactometer, fourthly the measurement set-up and more general, the uncertainty associated with dynamic olfactometry. Some critical points also apply more generally than only for livestock odours.

1.8.1 Odour panel selection and reference gas

Dynamic olfactometry is applied with qualified human assessors, the odour panellists. The panellists are crucial for the quality of odour measurements (Mannebeck, 2017). The odour panel is selected through performance tests with the reference odorant, n-Butanol. The aim of the panel selection is to attain a reliable sensor for regular odour measurements by selecting persons with an olfactory sensitivity within a predefined bandwidth (much narrower than the natural variability within the population) and which show a sustained individual repeatability (olfactory responses that are as constant as possible) from day to day and within a day (CEN, 2003). Therefore at least 10 individual threshold estimates (ITE) are determined per candidate for n-butanol and this over at least 3 sessions on non-consecutive measuring days. The results of the candidates for the odour panel are tested against the following criteria:

- Crit. 1: the individual repeatability: 'the antilog of the standard deviation s_{ITE} calculated from the logarithms (log_{10}) of the individual threshold estimates, expressed in mass concentration units of the reference gas, has to be less than or equal to 2.3'
- Crit. 2: the individual sensitivity: 'the geometric mean of the individual threshold estimates, expressed in mass concentration units of the reference gas, has to fall between 0.5 times and 2 times the accepted reference value for that reference material (for n-butanol this ranges from 62 µg/m^3 to 246 µg/m^3, or 0.020 µmol/mol to 0.080 µmol/mol).

These are important criteria to be verified, because a large variation in the population's sensitivity exists (Figure 1.8). The olfactory sensitivity of the general population can be represented by a typical bell curve as presented in Figure 1.13 (Nicolai et al., 1997).

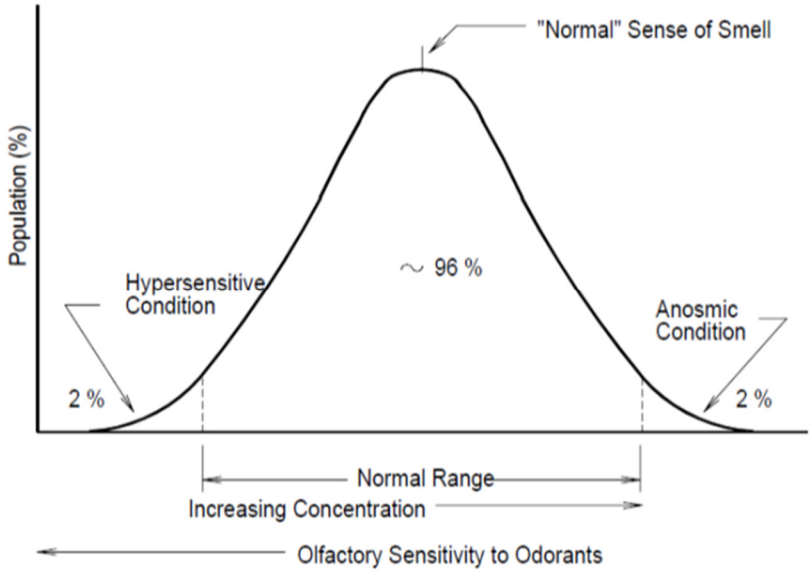


Figure 1.13 Distribution of olfactory sensitivity within the population (Source: Nicolai et al., 1997).

The large differences in human perception of the odour environment (Keller et al., 2012) include as well inter-individual differences (i.e. differences between individuals) as intra-individual differences (i.e. variation in sensitivity of the same person):

(a) According to Keller et al. (2012), inter-individual differences in olfactory acuity (perception) can be general (for different stimuli) or specific (related to specific odours). A large human olfactory psychophysics study (including 391 adults and 66 different odours) by Keller et al. (2012) showed high correlations between general olfactory acuity and factors such as race, gender, age, body type and smoking habits. The olfactory acuity declines with age and is generally lower in the male population (Keller et al. 2012). Odour-specific differences in olfactory perception were influenced by race, gender and age (Keller et al. 2012). Also previous experience with the specific odours and variability in the gene for the respective odorant receptor affect the odour-specific differences (Keller et al., 2012). Different human assessors can therefore estimate the odour concentration of the same odour sample differently (Brattoli et al., 2011, Gostelow et al., 2001).

(b) The olfactory sensitivity of a person can also vary in time because of physiological conditions (e.g. hormonal changes, illness, etc.), environmental and climatological conditions (e.g. a change in humidity) (Hangartner, 1985). A decreased general olfactory acuity can be due to infections, a trauma, exposure to toxic agents, neurodegenerative diseases, etc. (Keller et al., 2012). The high within-individual variability in olfactory psychophysics (the same olfactory stimulus can be perceived differently by the same person on different occasions) could be of the same order of magnitude as the variability between individuals (Keller et al., 2012).

To overcome these variations in sensitivity, n-butanol is used as reference material and for panel selection purposes, based on the following assumption as stated in CEN (2003).

CEN (2003) assumes in §3.3 that *"the sensitivity (of qualified panellists) for the reference will be a predictor for (their) sensitivity to other substances"*. This is an important asset of the reference gas because it implies traceability of odour units of any odorant to a defined amount of the reference gas. As indicated in section 1.6.1, the European odour unit (ou_E) is that amount of odorants that, when evaporated in $1m^3$ of neutral gas at standard conditions, produces the same physiological response of a panel as that of 1 European Reference Odor Mass (EROM), evaporated in 1 m^3 of neutral gas at standard conditions (CEN, 2003). 1 EROM of n-butanol corresponds with 123 µg of n-butanol

which, when evaporated in 1 m^3 of neutral gas at standard conditions, elicits a detection threshold from a panel qualified according to CEN (2003) and has a concentration of 1 ou_E/m^3 (CEN, 2003).
CEN (2003) specifies in §5.1. that *"the performance characteristics as determined on reference materials are transferable to other odours"* and with that regard indicates that *"when a laboratory fulfils the overall sensory quality criteria of trueness and precision (repeatability) for the reference material (i.e. n-butanol) set by CEN (2003), this quality level of performance is transferable to other environmental odours"* (Van Harreveld & Heeres, 1995; CEN, 2003), for example H_2S (McGinley & McGinley, 2010).

Of outmost importance is that the validity of these assumptions of CEN (2003) regarding n-butanol are being questioned by odour experts (McGinley & McGinley, 2010; Laor, Parker, & Page, 2014; Parker, Rhoades, Schuster, Koziel, & Perschbacher-Buser, 2005; Klarenbeek, Ogink, & van der Voet, 2014). Specifically, McGinley and McGinley (2010) reported that researchers are questioning the validity of the predictability assumption of CEN (2003). Since other (environmental) odours are experienced more regularly, researchers question the validity of panellists' selection using only one reference odorant (McGinley & McGinley, 2010). Laor et al. (2014) stated that selecting of panellists based on their sensitivity for the reference odorant does not necessarily mean that these selected panellists will also show a similar (average) sensitivity to other odorants. Based on several years of experience with odour measurements, Parker et al. (2005), indicated that n-butanol sensitivity is often weakly correlated to feedlot odour sensitivity. Klarenbeek et al. (2014) found a significant difference in variance components (for laboratory, panel and panel session) between n-butanol and different types of field odours, while working with EN13725-accredited laboratories, leading them to question the transferability assumption of CEN (2003). These concerns about the indicative character of the current reference gas has led to quests for alternative reference gases. The use of hydrogen sulphide and coffee odour as reference material have been tested (McGinley & McGinley, 2010; Mannebeck & Mannebeck, 2001). Hydrogen sulphide is now used in Germany as a complementary reference material during panel selection (personal communication, Olfasense). A few studies investigated the use of odorants' mixtures with similar properties as pig odour (Defoer & Van Langenhove, 2004; Qu & Feddes, 2007), because the characteristics of n-butanol differ from those of typical livestock odours (Qu & Feddes, 2007). Even CEN (2003) suggested to seek for a reference odour mixture to improve the accuracy and precision of olfactometric measurements. Klarenbeek et al. (2014) suggested based on their results to move

towards different reference substances or mixtures, depending on the characteristics of the odour source to be analysed. So far, no other reference odour mixture has been set.

The panel selection was introduced to decrease the variation in odour measurements and to obtain a reliable sensor. In practice, performing odour panel selection with n-butanol results in a low number of qualified panellists. According to Munoz et al. (2010), 50 up to 70% of potential panellists are excluded during panel selection. This poses a practical problem for smaller odour laboratories, which do not have the same financial capacity as bigger companies. Since it is questioned that n-butanol is sufficiently representative for other environmental odours, it means that a lot of potential panellists could be unnecessarily disqualified. A possible cause of this low success rate could be that only a few receptors of the human nose can detect n-butanol and malfunctioning of one of these receptors could cause specific anosmia for n-butanol (www.olores.org), leading the candidate to be insensitive to n-butanol.

Capelli et al. (2010) concluded that conducting panel selection and verification measurements one must take into account that expectation, habituation and motivation of the human assessors influences their results. They found a strong correlation between the initial dilution factor set by the operator and the measured odour concentration during panel selection and verification procedures. Panel selection is normally carried out starting from a known concentration of 60 ppmv of n-butanol. Considering the sensitivity range of qualified panellists, the set initial dilution step is mostly a factor of 8192. According to Capelli et al (2010) panellists then tend to indicate the presence of odour after 3 to 4 sample presentations by experience and thus independent of their real olfactory perception. This could reveal unfulfilling sensitivity during proficiency testing with n-butanol (Capelli et al., 2010). Capelli et al. (2010) therefore introduced a more selective procedure to deal with the “smartening” of assessors, namely starting from three different concentrations of n-butanol, unknown to the operator, to perform the panel selection and verification tests. This prevented the panellists from guessing or expecting the right answers (in order to keep their status as a panel member) and resulted in a more refined odour panel.

Of main importance is also the training of panellists despite the low attention given towards panellists’ training in CEN (2003). CEN (2003) states that new assessors must first be trained by performing at least one single measurement from which the results are discarded for the panel selection calculations. In literature evidence is found of the importance of panel training in order to achieve good assessors. Proper training of the

panellist can improve the reproducibility of the panellist results (Nicolai et al., 1997; Wihnen, 1986). Panellist experience is important to achieve stable individual thresholds (Laska & Hudson, 1991). A learning curve was observed for panel members who participate regularly to odour measurements by Clanton et al. (1999). According to Brattoli et al. (2011), panel members should be constantly screened and trained.

More detailed training procedures than prescribed by CEN (2003) are found in literature. McGinley and McGinley (1999) wrote that the training of new assessors should involve a training of olfactory awareness, sniffing techniques, standardized descriptors and olfactometry responses. They stipulate that assessors need to attend supplemental and re-certification courses. For the "Aerial Pollutant Emissions from Animal Confinement Buildings (APECAB) project, panellists were trained to apply appropriate breathing and sniffing techniques to intensify the contact between their olfactory sense and the odour sample. Also a standardized procedure was used, which consisted of numerous hours of training, to get repeatable results from each tested person (Jacobson et al., 2008). Also a more extensive training procedure is applied for the sniffing team method (Laor et al., 2011). As the quality of odour measurements relies on the reliability of the sensor, a more extensive training procedure for odour panels would be advised in CEN (2003).

According to Mannebeck (2017), next to the requirements for panel selection, set by CEN, the quality of an olfactometric assessment is largely influenced by panel member motivation (working atmosphere, payment), identification with the work (main task as a panel member in a company),… . The work of a panel member should not be underestimated, because it requires a high degree of concentration.

1.8.2 Odour sampling and storage

During the odour sampling the composition of the sample and the concentration of individual compounds can alter by interactions of the livestock odorants with the sampling material (bag, tubing, valve). CEN (2003) therefore states that polytetrafluoroethylene (PTFE, Teflon), fluorinated ethylene propylene (FEP, Teflon), polyvinylfluoride (PVF, Tedlar), polyethyleneterephthalate (PET, Nalophan), stainless steel and glass should be used as sampling material, because they tend to be "inert". A few studies have investigated the effect of the sampling inlet valve on sulphur compounds. In the study by Kim et al. (2006) it was shown that a stainless steel valve resulted in a higher loss of sulphur compounds compared to a Teflon valve (e.g. 45 % of loss of the most reactive odorant, H2S, when using a stainless steel valve, compared to

an insignificant loss when using a Teflon valve). In the same study, it was demonstrated that a long sampling time (5-10 min) resulted in a higher loss compared to fast sampling time (< 1 min).

The storage of odour samples preceeding the analysis in the laboratory, also influences the chemical composition (Trabue et al., 2006; Koziel et al., 2005; Sulyok et al., 2001; Guillot & Beghi, 2008; Mochalski et al., 2009; Hansen et al., 2011) and the odour concentration of the samples (Parker et al., 2010; Van Harreveld, 2003). CEN (2003) has defined that odour samples can be stored for up to 30 h, which allows the sample to be collected one day and transported to the laboratory for analysis the next day. Three types of sampling bag materials can be used for dynamic olfactometry including, fluorinated ethylene propylene (FEP, Teflon), polyvinylfluoride (PVF, Tedlar) and polyethyleneterephthalate (PET, Nalophan). The recovery of agricultural odorants (sulphur compounds, carboxylic acids, phenols and indoles) in these types of sample bags has been investigated in a number of studies (Trabue et al., 2006; Koziel et al., 2005; Sulyok et al., 2001; Guillot & Beghi, 2008; Mochalski et al., 2009; Hansen et al., 2011). In general these studies demonstrate that all odorants are lost to some extent during 24 h of storage and the degree of recovery is for sulphur compounds (65-95%) > carboxylic acids (30-70%) > phenols/indoles (5-25%). A study with PVF bags demonstrated that the recovery of trimethylamine after 24 h was approximately 50% (Wzorek et al., 2010). There could be different reasons for the loss of odorants during storage including, adsorption to the bag material, diffusion through the bag walls and reactive losses (Van Durme & Werbroeck, 2015). Other factors such as sample bag volume and initial concentration may also influence the recovery. A low initial concentration is more sensitive to adsorption processes and a small bag volume is more sensitive to diffusion processes due to a large surface area relative to the bag volume. It has also been demonstrated that the recovery of odorants is affected immediately after filling the bags (Hansen et al., 2011) or within the first 0.5 h (Trabue et al., 2006; Koziel et al., 2005). This means that the chemical composition and the odour concentration can alter, even when the samples are analyzed within the first hours after sampling. Since sample storage affects the chemical composition of odour mixtures to such an extent, it is important that a fixed duration in between sampling and olfactometric analysis is set in order to obtain repeatable results between different laboratories (e.g. to evaluate replicate samples of odour abatement techniques.

It has been demonstrated that particularly PVF bags can have a large background impurity especially of N, N-dimethylacetamide and phenol (Trabue et al., 2006; Koziel et

al., 2005; Parker et al., 2010; Bokowa, 2012), whereas FEP and PET bags have low background impurities (Koziel et al., 2005). In the study by Bokowa, (2012) it was demonstrated that a new PVF bag filled with nitrogen can have an odour concentration between 100-130 OU_E/m^3, but continuously flushing and heating at 100 °C can reduce the background to less than 1 OU_E/m^3 within 36 h. In the study by (Trabue et al., 2006) it was also demonstrated that three bag fillings with odorous air decreased the off-gassing of acetic acid and phenols from PVF bags, but the compounds were still elevated compared to the input concentration in the odorous air. It appears that FEP and PET bags should be preferred for dynamic olfactometry since these bags have low background impurity. The background odour from new, non-flushed Tedlar and Nalophane bags, in which fresh air was stored for 24h, was 75-317 odour units/m^3 in case of Tedlar (FEP) and 36-43 odour units/m^3 for Nalophane (PET) (Laor et al. 2010). This could be reduced by preflushing the bags towards 25-32 odour units/m^3 in case of Tedlar and to 19-22 odour units/m^3 in case of Nalophane (Laor et al. 2010). However, all three bag types result in compound losses during storage and this has to be taken into account when the results are evaluated.

In relation to animal production, dynamic olfactometry is often used to evaluate the performance of an abatement technology. It is difficult to predict to what extent sample loss due to adsorption on the bag walls and permeation through the bag walls will affect the efficiency measurement of the abatement technique. The effect of adsorption is more likely to be seen on samples taken at the outlet of the abatement technique, since they tend to have a lower odour concentration. Permeation or diffusion through the bag walls is a slow process and can affect both bags, but the extent to which the sample will be affected, will not only depend on the initial concentration in the bags, but will also depend on the duration of sample storage. It is however inherent to olfactometry, that samples are transported and stored for analysis, since it is practically difficult to set up a mobile odour laboratory according to the laboratory requirements described in CEN (2003).

CEN (2003) does not include a quantification of the effects of the sampling material and storage time on the chemical composition or odour concentrations of samples. It sets a maximum time interval between sampling and analysis of up to 30 h in order to minimize adsorption, desorption, etc. and suggested that the above mentioned materials would be used to minimize interactions between the sampled odour and the sampling material.

The CEN standard allows for filtration of airborne dust with a particle filter to protect the olfactometer. The effect of this procedure on the odour measurement will depend on the extent of gas/particle partitioning of the odorants constituting the odour sample. Although

odorants have been detected in particles (Cai et al., 2006; Bulliner et al., 2006), systematic information on gas/particle partitioning is very scarce. The study by Andersen et al. (2014) in which adsorption to sampling filters was accounted for, showed that, in general, odorants are mainly present in the gas phase. Only carboxylic acids with more than 4 carbon atoms partitioned to a significant degree (>10%) into the particle phase. Based on this, the key livestock odorants, identified by Hansen et al. (2012), are predicted to be predominantly present in the gas phase. This is in line with the rough estimation by Hammond et al. (1981) that gas phase odorant mass is around 4-5 times higher than particle odorant mass. Walgraeve et al. (2015) more recently showed that, although particles present in a pig house are enriched with VOCs, the fraction of adsorbed volatiles is very low: less than 0.11 % for the studied compounds (acetic acid, butanoic acid, phenol, dimethyldisulphide) and considering the particulate fraction PM10 at a concentration of 1 mg m^{-3}. Therefore the loss of odorants in dust through sample filtration can only influence the odour concentration measurements to a small extent.

1.8.3 Interactions of livestock odorants during analysis

CEN (2003) (CEN, 2003) formulates different requirements for the measuring equipment. The parts that come in contact with the odourous sample, shall be constructed from materials which (1) are odourless, (2) minimize physical and chemical interaction with the odourous sample, (3) have a low permeability in order to minimize loss due to diffusion and (4) have a smooth surface (CEN, 2003). The length and diameter of internal tubing should be minimized to avoid contamination of the olfactometer by the odorants and also the residence time of the odorous sample in the olfactometer should be limited to prevent contamination of the olfactometer (CEN, 2003). Orifices should be large enough to prevent blockage by particulate contamination (CEN, 2003). The use of hot-wire anemometers or other devices that change the gas characteristics are not allowed by CEN (2003). Devices which influence the characteristics of the odourous sample, for example its temperature, should also be avoided in the construction of the olfactometer (CEN, 2003). The olfactometer must be designed in such a way that noise or other stimuli cannot reveal the location or concentration of the odorant (CEN, 2003).

In practice, materials such as glass, Teflon and stainless steel, as advised by CEN (2003), are often used for the construction of an olfactometer. Some studies however show concern with the influence of the olfactometer on the recovery of odorants after

passage through the dilution system. In the study by Hansen et al. (2010) the recovery of sulphur compounds was evaluated, because these volatile sulphur compounds are key compounds of intensive livestock odour (Mackie et al.,1998; Feilberg et al., 2010; Hansen et al., 2012). Two types of olfactometers, one constructed by stainless steel/Teflon and one with glass, respectively were tested. The recovery of hydrogen sulphide, methane thiol and dimethyl sulphide ranged between 50 to 90% for both types of olfactometers. In another study by Hansen et al. (2013) the recovery of a wider range of odorants including sulphur compounds, carboxylic acids, trimethylamine, m-cresol (a surrogate for p-cresol) and n-butanol, typical for swine facilities (Ni et al., 2012), were evaluated using the stainless steel/Teflon olfactometer. Hansen et al. (2013) demonstrated that all odorants were affected by the olfactometer to some extent, except for dimethyl sulphide. Hydrogen sulphide, methane thiol and n-butanol were not completely recovered but these compounds reached a stable level in the outlet from the olfactometer within a few seconds. Carboxylic acids, trimethylamine and m-cresol were more affected by the olfactometer. Even during a 60 s pulse duration, these compounds did not reach a stable concentration level in the outlet. Since odorants from animal production are highly volatile and are normally found in the low ppbv-range, the authors concluded that diffusion, adsorption and reactive losses of odorants could have a large influence on the measuring results. Kasper et al. (2017) recently tested the recovery of a selection of livestock odorants (hydrogen sulfide, methane thiol, dimethyl sulfide, acetic acid, butanoic acid, propanoic acid, 3-methylbutanoic acid, 4-methylphenol, and trimethylamine) and of n-butanol in three different olfactometer dilution systems (1. A custom-built with glass tubes, 2. A TO8-olfactometer, 3. An olfacton dilution system based on a mass flow controller) providing from accredited laboratories with PTR-MS. The overall recovery of the tested livestock compounds and the variability between dilution levels was different for the three types of olfactometers (Kasper et al., 2017). The degree of recovery depended on the odorant type (different behaviour in the olfactometer system) and the pulse duration (2.2 s, 15s and 60s were tested) (Kasper et al., 2017). In case of 2.2 seconds of pulse duration (corresponding with pulse duration of Yes/No presentation mode in TO8), the average recovery over different compounds of the glass olfactometer was 51.0 %, while for the TO8 (Germany) this was 69.5 % and for the Olfacton (the Netherlands) this was 52.0 % (Kasper et al., 2017). After 15s resp. 60 s of pulse duration, the average recovery in the glass olfactometer was 72.3% resp. 76.0%; in the TO8 it was 77,8% resp. 91.8%; in the Olfacton that was 80,1% resp. 88.8% (Kasper et al., 2017). Striking was the adsorptive behaviour of the reference gas n-butanol, used for panellists' selection and the different behaviour in the different olfactometer types (Kasper et al., 2017). The recovery was 33.9% (2.2s), 62.2% (15s), 61.7% (60s) in the

glass olfactometer, while 68.1% (2.2s), 76.7% (15s), 98.8% (60s) in the TO8 and 29.5% (2.2s), 70.0% (15s), 77.6% (60s) in the Olfacton (Kasper et al., 2017). The adsorption of n-butanol also depended on the dilution level (Kasper et al., 2017). In the Olfacton, the degree of recovery of n-butanol was only 9.4% at the highest dilution level, the degree of recovery slowly increased towards 81.8% at the lowest dilution level (Kasper et al., 2017). They concluded that stainless steel and glass should not be used in sampling equipment and that the use of PFA, PTFE and SilcoTek-coated steel is advised. To improve the reproducibility between laboratories, further standardization is advised (Kasper et al., 2017).

Next to the fact that odorous compounds can adsorb onto the contact surfaces of the olfactometer, namely on the tubing and on the dilution system (Jiang, 2006), cross-contamination of odour samples can occur as a result of odorant adsorption on the tubing and on the dilution system and can also result in bias (Munoz et al., 2010).

1.8.4 Setup of the test procedure

As mentioned by Gostelow et al. (2001) and Brattoli et al. (2011), the setup of the test procedure is a one of the key variables in olfactometry, which influences the measurement result. Within the boundaries indicated by CEN (2003), odour labs have a certain freedom in the measurement protocol they apply. Sources of variation in the setup of the test procedure are the panel size, the presentation and choice method of the olfactometer, the number of dilution series, the start step of the dilution series,… Their influence will be addressed further in this chapter.

1.8.4.1 Panel size

Following CEN (2003), the panel should at least count four panel members after retrospective screening for a valid odour concentration determination. However, a larger panel size is recommended to enhance the repeatability limit and the accuracy of the measurement result (CEN, 2003).

Only a few studies related panel size to olfactometry performance. Van Harreveld and Heeres (1995) performed an investigation in the pre-CEN period using n-butanol only and chose that a panel size of 5 would be used and 3 rounds, of which the first systematically was discarded, was preferred.

Mannebeck (2017) advises that more and less sensitive panellists, fulfilling both CEN selection criteria, are uniformly distributed in a panel when performing odour measurements.

1.8.4.2 Sample presentation

1.8.4.2.1 Choice mode

Two methods exist for the presentation of dilutions and the choice mode for the panellists, namely the Yes/No and the Forced Choice Method (CEN, 2003, Brattoli et al., 2011). CEN indicates that both methods produce an individual threshold estimate (ITE) as a common result and that the use of the ITE derived from either of these methods is identical. In the Yes/No mode, the assessor is asked to evaluate the gas coming from one specific port and to point out whether an odour is perceived or not. The assessor presses the yes-button in case an odour is perceived. In the Yes/no-mode, the assessor knows that sometimes blanks (neutral gas) are presented. In the Forced choice mode the assessor is presented with two or more ports and the assessor has to decide through which port the odour stimulus is presented. At the same time, the other port presents the neutral gas (odourless air). The location of the odour stimulus in successive presentations is randomly spread over the available ports. The assessor must indicate which of the ports presents the odour stimulus. When the assessor is in doubt, he/she must designate a port 'at random' and must indicate whether this choice was a guess, an inkling or a certainty. (CEN, 2003) Only correct indications of which the assessors were certain, are considered in the calculation of the result (CEN, 2003). There are also “mixed” methodologies e.g. in some cases the yes/no method is used but the reference gas is presented as well (from another port). CEN assumes that the results of both choice modes are comparable There are only a few reports on the comparison between “yes/no” and the “forced choice” approach, written by Van Boheemen, 2012; Maxeiner, 2006; Maxeiner, 2007. Based on these reports, no statistically significant differences could be noticed between the results of the “yes/no” and the “forced choice” approach.

1.8.4.2.2 Dilution series

The number of dilution series and the panel size both influence the precision of the odour measurement (CEN, 2003). According to CEN, at least 2 valid dilution series or rounds are required to determine a panel threshold. It is allowed to perform 3 rounds including a preliminary round from which the results are systematically discarded (CEN, 2003). Apart from the pre-CEN studies of Van Harreveld (1995) and Clanton (1999), the effect of the number of dilution series on the measurement result has not been quantified.

According to Clanton (1999) the panel size however would have more effect than the number of dilutions' series on the precision: variance was reduced applying one dilution series with eight panellists rather than performing two dilution series with four panellists.

Also the starting step of a dilutions' series proves to be important. Capelli et al. (2010) found a strong correlation between the measured odour concentration and the first dilution factor at which the odour sample was presented to the panellists.

According to CEN (2003), at least 3 and rather 5 or 6 presentations are required within one dilutions' series. When the Yes/No mode is applied, every dilution series should include at least one blank at a random position. The forced choice method always delivers a zero reference so no extra blanks should be presented. Equally important is to not make a dilutions' series too long, to prevent olfactory fatigue or adaptation of the assessors towards the odour under investigation.

The different presentations in one dilution series will be given in either an ascending or random order of stimuli (CEN, 2003). An ascending order of presentation is preferred (Brattoli et al., 2011; Gostelow et al., 2001), which means that higher dilutions are presented before lower dilutions in order to prevent the influence of adaptation of panellists towards the odour or olfactory fatigue on the measurement results (Gostelow et al., 2001). It also could minimize adsorption and desorption problems in the olfactometer (Gostelow et al., 2001; Brattoli et al., 2011). The disadvantage of using a strict order of dilutions (e.g. an ascending order), is that panellists will start to expect successive presentations to become stronger and that they can adapt their response accordingly (Gostelow et al., 2001). Olfactory fatigue and adsorption/desorption problems however, have a larger impact than the anticipation effect (Gostelow et al., 2001). According to Mannebeck (2017), using the yes/no mode and an ascending order of stimuli an average panel could reliably measure about 16 samples per day, if every 4 to 6 samples are followed by a break of at least 30 minutes.

The step factor in between consecutive dilution steps influences the precision of odour measurements. CEN allows a step factor between 1.4 and 2.4 (inclusive) between consecutive dilution steps, which has to be kept unchanged throughout the measurement. According to Ogink et al. (1995), the step factor (comparing 1.4, 2.0 and 3.0) had a significant influence on the precision of odour measurements (higher precision with a lower step factor): Considering a panel size of 8 panellists and a factor of 1.4 in between consecutive dilution steps, the repeatability factor (section 1.8.5.2) was 1.7; while for a step factor of 2.0, which is common in olfactometers, the repeatability factor

was only 2.26 and considering a step factor of 3.0 in between the presented dilutions the repeatability factor increased to 3.34, which is higher than allowed by CEN(2003) (limit is a repeatability factor of 3). Also the bias reduced with a lower step factor (Ogink et al., 1995). Boeker et al. 2007, 2008, however only found a minor influence of the dilution factor on the measurement uncertainty, evolving from 4.4 dB towards 4.5 dB when the dilution factor was varied from 1.4 towards 2.4.

1.8.4.2.3 Air flows at the sniffing ports

Different flow rates at the sniffing port can have an important influence on the determined odour concentration (Gostelow et al., 2001). According to CEN (2003), the air flow from a sniffing port shall be at least 20 L/min. Clanton et al 1999 studied the effect of air flow rate and found a difference of 9 % to 28 % in odour units for the same sample (3 sample strengths of pig house odour analysed), using two different air flow rate calibrations.

1.8.5 Measurement uncertainty

1.8.5.1 Olfactometric variability

Experts agree that a high amount of uncertainty is related to olfactometry measurements (Boeker et al., 2008; Boeker, 2014; Zarra et al., 2008; Zarra et al., 2009; Andersen, 2013).

In general, the measurement uncertainty is a parameter, related to a measurement result, that quantifies the dispersion of values that could realistically be attributed to the measurand (Higuchi, 2009).

The main source of uncertainty of the olfactometric method is the inherent variability of the human sense of smell (Boeker et al., 2008; Zarra et al., 2008), The sense of smell is highly variable, both in time and among individuals (Boeker, 2014; Laor et al., 2014; Zarra et al., 2008; McGinley & McGinley, 2006; Clanton et al., 1999).

Five forms of variability can be distinguished (Laor et al., 2014):

- Intra-panellist variability: the variability of an individual panellist over time, when repeating the measurement of the same sample (Van Harreveld & Heeres, 1997; Clanton et al., 1999; Laor et al., 2014).
- Inter-panellist variability: the variability in sensitivity between different panellists, whom measure the same sample (Van Harreveld & Heeres, 1997; Clanton et al., 1999; McGinley & McGinley, 2006; Laor et al., 2014).
- Intra-panel variability: variability can occur within the same odour panel as the panel repeats the measurement of the same sample over time (Clanton et al., 1999; McGinley & McGinley, 2006; Klarenbeek et al., 2014; Laor et al., 2014).
- Inter-panel variability: variability can arise between the results of different odour panels in a single laboratory when evaluating the same sample (Clanton et al., 1999; McGinley & McGinley, 2006; Klarenbeek et al., 2014; Laor et al., 2014).
- Inter-laboratory variability: variability can arise when different laboratories analyse the same sample (McGinley & McGinley, 2006; Klarenbeek et al., 2014; Laor et al., 2014).

Table 1.13 presents estimations of these five forms of variability based on literature. The number of values present in literature however are limited.

Table 1.13 Estimations of the variation occurring at different levels during measurements based on literature

Variance level	Variation measured between repetitions	Tested odour
Intra-panellist	Up to a factor 4 of difference in time [1]	Swine odour
Inter-panellist	A factor 4 of difference (in sensitivity) is possible between the detection thresholds of selected panellists. (Crit. 2, CEN (2003)) [2]	n-Butanol
	Up to a factor 7 of difference between panellists [1]	Swine odour
Intra-panel	An estimated, average (n=27) repeatability factor of 1.4 between consecutive repetitions of the same sample by the same panel [3]	n-Butanol and environmental odour
Inter-panel	An estimated, average (n=50) repeatability factor of 1.92 [3]	n-Butanol
	Repeatability factors of individual laboratories between 1.84 and 12.9 [4]	n-Butanol
	An estimated, average (n=25) repeatability factor of 1.50. Up to a factor 1.77 of difference between the detection thresholds of two panels [3]	Environmental odour
Inter-laboratory	Up to a factor 3 of difference between the panel thresholds of three laboratories [5]; Factor 4.3 [7]	n-Butanol
	Up to a factor 7 of difference between three laboratories [5]	Swine and sow odour
	Up to a factor 50 of difference between three laboratories [5]	Dairy odour
	Up to a factor 27 of difference between the mean concentration of 3 laboratories [6]	Pig odour measured at a biological air-cleaner
	Factor 6.3 [7]	Field odours

1 Clanton et al. (1999): individual laboratory assessment

[2] Laor et al. (2014) based on CEN (2003)

[3] McGinley and McGinley (2006)

4 Maxeiner (2006)

5 Bereznicki et al. (2012)

6 Jonassen et al. (2012)

7 Klarenbeek et al. (2014)

Variability does not only exist at the measurement level, also the sampling procedure and sample storage introduce variation in the results (see section 1.8.2). Laor et al. (2010) reported average losses in odour (concentration) of a factor of 6 (Range: 4.61-6.75) for calves' manure odour sampled in Tedlar bags after a storage time of 24h and on average by a factor of around 2 (Range: 1.16-2.67) when sampled in a Nalophane bag. Up to a factor 13 of difference in odour detection threshold (for an automotive facility) was found by Bokowa (2010) depending on the sampling method (predilution or not).

Also the accuracy (which cannot be assessed for real samples) and the data-analysis method (American vs. European standard) influence the uncertainty on olfactometric results. According to Boeker et al. 2008, the scores of the panellists for the panel selection criteria of CEN (2003) can have a large influence on the measurement uncertainty associated with dynamic olfactometry.

All these sources of variability contribute to the large uncertainty of the complete olfactometry assessment (Boeker et al., 2010; Laor et al., 2014).

When evaluating the odour emissions of livestock facilities, also the measurement of the ventilation rate is a source of uncertainty (Ubeda et al., 2013). Model errors and meteorological parameters also influence the uncertainty of an odour impact assessment (Ubeda et al., 2013), but the uncertainty on the olfactometric measurement is considered to be larger.

1.8.5.2 CEN (2003) addresses laboratory performance requirements

The measurement uncertainty as such is not addressed in CEN (2003) (Boeker et al., 2008; CEN, 2003). The standard CEN (2003) however prescribes two quality requirements for laboratory performance. They are imposed to ensure reproducible results and to minimize measurement uncertainties of the olfactometric method (Mannebeck, 2017). These quality requirements consist of an accuracy limit and a precision limit (CEN, 2003; Capelli et al., 2010). An individual laboratory can test compliance to these requirements by performing at least 10 odour measurements with the reference gas, n-butanol during a year and by calculating its' precision and accuracy limit as follows (CEN, 2003; Capelli et al., 2010):

First, the precision in terms of ***repeatability*** can be assessed. The repeatability standard deviation s_r therefore is calculated using the formula:

$$s_r = \sqrt{\frac{\sum_{i=1}^{n}(y_i - \bar{y}_w)^2}{n-1}} \quad \text{(Eq. 1.1)}$$

where: n is the number of test results, $\bar{y}_w$ is the average of the test results and y_i is the test result.

The repeatability limit *r* represents the maximum difference between two consecutive repetitions of the same sample by the same laboratory found in 95 % of the cases. It can also be expressed as its' antilog, namely the repeatability factor (10^r). The repeatability limit r can be calculated using:

$$r = t.\sqrt{2}.s_r \quad \text{(Eq. 1.2)}$$

where: t is a factor from the Student's t-distribution for $n-1$ degrees of freedom and a 95 % -confidence level.

The repeatability standard deviation (s_r) and repeatability limit (r) have to comply with:

$$s_r \leq 0.1721 \quad \text{(Eq. 1.3)}$$

$$r \leq 0.477 \text{ or } 10^r \leq 3 \quad \text{(Eq. 1.4)}$$

This requirement means that the difference between two consecutive single measurements on the same testing material in one odour laboratory under **repeatability conditions** should not be larger than a factor 3 in 95 % of the cases (CEN, 2003).

In that perspective, CEN (2003) §5.1 describes that since olfactometric measurements take a relatively long time, a series of repeated measurements can stretch over more than one day (CEN, 2003) and due to practical considerations, the composition of the

panel can alter in between sessions. These variations are common in the olfactometric practice and therefore are accepted and considered to fall within repeatability conditions (CEN, 2003).

The second quality parameter is the ***accuracy*** of the odour concentration (A_{od}). The accuracy encloses both the trueness (expressed in terms of bias, caused by systematic error) and the precision (random error). The trueness ($|d_w|$) is expressed as the estimate of the within-laboratory bias.

The within-laboratory bias d_w is calculated by:

$$d_w = \bar{y}_w - \mu \quad \text{(Eq. 1.5)}$$

Where: $\bar{y}_w$ is the average of the test results and μ is the accepted reference value (after $\log_{10}$ conversion).

The accuracy A_{od} can then be calculated using:

$$A_{od} = |d_w| + (A_w.r) \quad \text{(Eq. 1.6)}$$

Where: d_w is the within-laboratory bias, which reflects the trueness of the odour concentration; A_w is a statistical factor that is function of the number of test results and r is the repeatability limit.
The statistical factor A_w is calculated by:

$$A_w = \sqrt{\frac{1}{2.n}} \quad \text{(Eq. 1.7)}$$

The accuracy of the odour concentration A_{od} should comply with:

$$A_{od} \leq 0.217 \quad \text{(Eq. 1.8)}$$

The within-laboratory bias cannot be assessed for non-reference materials (CEN, 2003), because there is no accepted reference value (CEN, 2003). As a consequence the accuracy cannot be quantified for non-reference materials. However, when inter-laboratory testing is performed, the geometric mean of the results of all laboratories for the environmental odour is usually considered as the best estimate of the reference value, enabling to make an estimation of the accuracy of the laboratory for the environmental odour (CEN, 2003). 10 test results for the environmental odour then are required to assess the accuracy of the individual laboratory.

Before calculating the accuracy and the precision limit, the test results (odour concentrations expressed in [ou_E/m^3]) undergo two transformations: they are first

converted into analytical concentrations [ppb] (calculated using the analytical concentration of the reference and the respective dilution factor) and secondly these analytical concentrations undergo a log_{10}-conversion (CEN, 2003).

The allowed accuracy and repeatability limit may seem very large to those not familiar with the variability associated to olfactometric measurements (Laor et al., 2014), however those limits are often not achieved in practice (Laor et al., 2014; Van Harreveld et al., 2009; Munoz et al., 2010; Ubeda et al., 2013). Interlaboratory comparison tests in the years 2005 till 2008 showed that only 20 up to 41 % of the participating laboratories fulfilled the performance criteria set by EN13725, analysing n-butanol (Maxeiner, 2006; Van Harreveld et al., 2009). In 2014, an interlaboratory comparison test on Chinese fish sauce odour was organised by Odournet Gmbh (Maxeiner, 2015) and only 1 of the 15 participating laboratories fulfilled both performance criteria of CEN (2003) for this real odour mixture.
CEN (2003) §5.1 stated that by setting an accepted reference value for the reference substance n-butanol, the reproducibility of results between laboratories is ensured implicitly. However the limited number of proficiency tests show strong interlaboratory differences, up to a factor of 50 (Bereznicki et al., 2012; Laor et al., 2014; Jonassen et al., 2012; Jonassen et al., 2014).

1.8.5.3 Measurement uncertainty estimations

The Guide to the Expression of Uncertainty in Measurement (GUM), published by the International Organization for Standardization (ISO, 1995), presents a methodology to assess the measurement uncertainty (Higuchi, 2009). According to this standard, the measurement uncertainty can be estimated statistically based on sufficiently reproduced measurements or based on a modelling of the measurement process, e.g. using a Monte Carlo simulation model.

Since no specific method is described in the CEN standard to calculate the measurement uncertainty on olfactometric results, Boeker et al. (2007) applied the different methodologies, described in GUM-method (ISO, 1995) to assess the measurement uncertainty on olfactometry.

The expanded uncertainty (U) was defined as:

$$U = k \times s \qquad \text{(Eq. 1.9)}$$

With s being the standard deviation and k an expansion factor of 2 to consider 95 % of the values instead of 68 %.

Boeker et al. (2007) chose to express the measurement uncertainty in odour decibels (chapter 1.6.1):

$$U\ [dB_{OD}] = 10 \times \log_{10}(c_{od\text{-}olf} / c_{od\text{-}real}) \qquad \text{(Eq. 1.10)}$$

With $c_{od\text{-}olf}$ being the odour concentration measured by dynamic olfactometry and $c_{od\text{-}real}$ the real odour concentration of the sample.

The expressed measurement uncertainty (Eq 1.10) thus was a measure for the possible ratio between the measured odour concentration and the real odour concentration of that sample.

Firstly, Boeker et al. (2007) calculated the expanded measurement uncertainty using the comparative standard deviation, established between CEN-accredited laboratories for n-butanol during interlaboratory comparison tests in 2003 and 2005 (Maxeiner 2003; Maxeiner 2006). An expanded uncertainty of measurement of 6 dBOD was deviated by Boeker et al. 2008, using the results of the ICO-tests. This corresponds with an error band between one forth and the fourfold of a measured value (Table 1.14).
Secondly, Boeker et al. 2007 set up a Monte-Carlo simulation model that simulated olfactometric measurements according to CEN (2003). The uncertainty was simulated for varying conditions in panel size, panellists' selection criteria (individual sensitivity and repeatability), step factor of olfactometer, with and without retrospective screening (Boeker et al 2007, 2008). A measurement uncertainty of 4,5 dBOD (Table 1.14) was found based on the Monte Carlo simulation model for the reference situation (panel size 4; step factor 2 between consecutive dilution steps; panellists with an individual repeatability of 2.3 according to panel selection criterion 1). Translated from the decibel scale to the level of odour concentrations expressed in odour units, this means an error band from one third to the threefold of an actual measured value (Table 1.14). In other words, for an olfactometric measurement resulting in an odour concentration of 2100 ou_E/m^3, the uncertainty would range from 700 ou_E/m^3 to 6300 ou_E/m^3.
According to Boeker et al. (2008), the results from the round robin tests lead to the best estimation for the uncertainty in practical measurements, because it also considers differences between laboratories next to laboratory-specific differences.

Table 1.14 summarises the results of the uncertainty calculations performed by Boeker & Haas (2007) and by Klarenbeek et al. (2014). The study performed by Klarenbeek et al. (2014) was based on measurements of n-butanol and non-butanol odours by 5 Dutch ISO/IEC 17025:2005 - accredited laboratories during proficiency tests in 2004-2012 organised in the Netherlands. An expanded uncertainty of 4.46 dB was found for n-butanol measurements and of 5.64 dB for non-butanol (environmental odour) measurements, corresponding with a possible ratio of 2.8 resp. 3.7 between the measured value and the real value of a sample for n-butanol resp. non-butanol odorants (Klarenbeek et al. 2014; table 1.14). In the study by Higuchi (2009), the uncertainty ratios on the odour index (a sensory index of odour measured using the triangular odour bag method; Environment Agency, 1995) varied between a factor of 3.1 and 6.7, using the results of interlaboratory comparison tests from 2003 till 2007, and they were depending on the analysed odour compounds. Higuchi (2009) stated that the expanded uncertainty was smaller for organic solvents compared to sulphur compounds since their psychophysical perception by the human nose is different.

Table 1.14 Estimations of the uncertainty on olfactometry (Boeker & Haas, 2007 [a]; Klarenbeek et al., 2014 [b])

Source of data	Measurement uncertainty	Corresponding ratio between measured and real value	95 % confidence interval around measured value
Interlaboratory comparison test (n-butanol) [a]	6 dB_{OD}	4	Between 1/4th and 4x measured value
Interlaboratory comparison tests (n-butanol) [b]	4.46 dB	2.8	Between 1/(2.8) and 2.8x measured value
Monte Carlo simulation (theoretical CEN-limit values) [a]	4,5 dB_{OD}	3	Between 1/3rd and 3x measured value
Interlaboratory comparison tests (environmental) [b]	5.64 dB	3.7	Between 1/(3.7) and 3.7x measured value

1.8.5.4 Implications for the interpretation of olfactometry results

The large measurement uncertainty on olfactometry complicates the interpretation of results by stakeholders, such as researchers, manufacturers of abatement techniques, environmental consultants, policy makers and environmental regulatory agencies, while a correct interpretation of results is important for farmers and their neighbouring residents (Ubeda et al., 2013; Higuchi, 2009; McGinley, 2002; Munoz et al., 2010).

Specifically, considering legislation limits of 0,5 – 5 ou_E/m^3 as 98-percentile, depending on the specific situation (an isolated farm versus a farm belonging to a cluster of farms) at the nearest resident (see chapter 1.6), it follows that the large variation associated with olfactometry can pose problems in the evaluation of permits.
Boeker et al. (2007) illustrated, in that view, that the uncertainty associated with dynamic olfactometry could be considered when evaluating the fulfilment of odour limits.
Boeker et al. (2007) (Figure 1.14) showed, as follows, how a measurement uncertainty of 6 dB could influence the evaluation of fulfilment of an upper limit odour concentration of 1000 ou_E/m^3:

- Between 250 ou_E/m^3 and 4000 ou_E/m^3, there is an 'indifference range' determined by the limit value and the interval of the measurement uncertainty (Boeker et al. 2007).
- When the measured odour concentration is lower than 250 ou_E/m^3, the predefined limit is met with 95 % certainty (Boeker et al., 2007). Only when the measured concentration is higher than 4000 ou_E/m^3 the predefined limit is exceeded with 95 % certainty (Boeker et al., 2007).

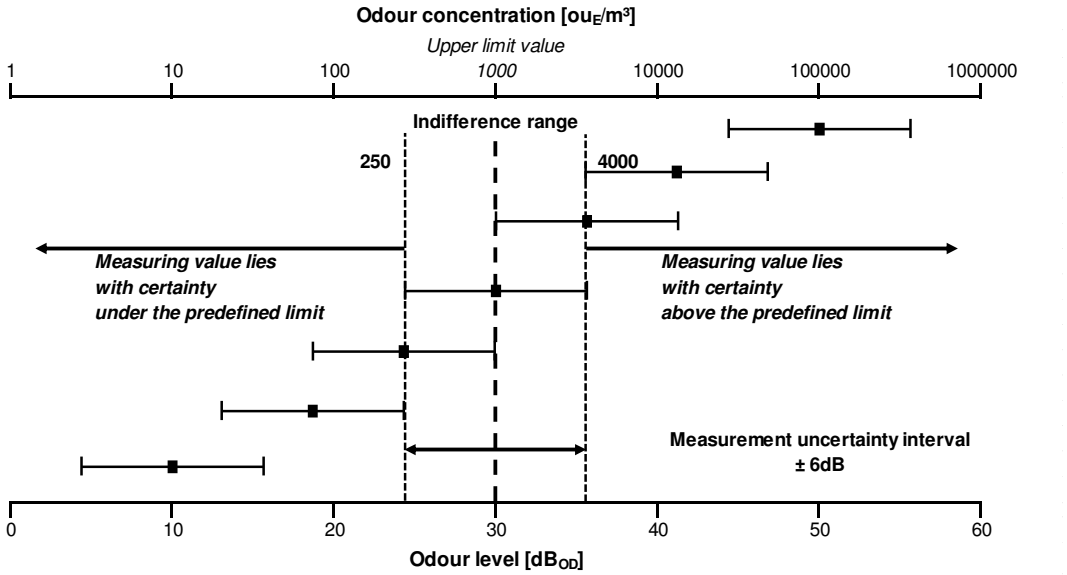


Figure 1.14 Example of the interpretation of an uncertainty of 6dB for a requested upper limit concentration of 1000 ou_E/m^3 (adapted from Boeker et al., 2007)

Boeker et al. (2007) suggested that when investigating conformity with odour limits, it should be stated which position is to be followed, because the measurement uncertainty could mean an intensification or reduction in requirements. Boeker et al. (2007) proposed when a complainant wanted to show an exceedance of the limit value, measured values above the limit value plus the measurement uncertainty could be considered as violations of the limit. They also suggested that when a farmer wanted to show compliance with a limit value, measured values below the upper limit minus the measurement uncertainty could be considered proving compliance.

The large measurement uncertainty is also problematic when evaluating odour abatement techniques. Jonassen et al. (2012) found a difference in reduction efficiency of a factor 5 (80 % efficiency, 18 % efficiency, 16 % efficiency) for between 3 laboratories. Laor et al. (2014) stated the uncertainty associated with dynamic olfactometry directly influences the uncertainty associated with the use of odour dispersion modelling and could eventually lead to an over- or underestimation of the impact radius of an emission source.

1.8.6 Summary of the literature review

In summary, the critical points in the application of CEN (2003) for livestock odour measurements are the loss of odorants from sampling bags during sample storage; the adsorption of agricultural odorants on the olfactometer's tubing during analysis causing an incomplete recovery of the sample at the nose masks; the large measuring uncertainty on olfactometry and the resulting low reproducibility between laboratories along with the questioned respresentativeness of the reference gas n-butanol.
The measurement uncertainty and the representativeness of the reference gas are thought to have a substantial impact on olfactometry results within and between laboratories.

1.9 Problem statement

Pig husbandry is an important economical and agricultural activity in Flanders. The expansion of pig husbandry from 2009 onwards has led to a growing awareness of its' impact on the air quality, especially while coinciding with a high population density. The government has the difficult task of working towards a balance between a sustainable pig production and ensuring a qualitative and healthy living environment for neighbouring residents of these farms.

Dynamic olfactometry, performed according to CEN (2003)-standard, is the most-commonly applied method in Europe, for the measurement of total odour emissions and to verify agreement with odour regulations. It is also largely applied to test the efficieny of odour abatement techniques. The large implications of olfactometric results on stakeholders, emphasise the importance of reliable and precise measurements. Nonetheless, different issues appear when applying CEN (2003) in odour assessments. Amongst them, the large measurement uncertainty on dynamic olfactometry and the resulting poor reproducibility between laboratories and also the questioned representativeness of the reference gas used for panellists' selection and which is the basis for traceability. To address these issues with regard to pig house emissions, it was necessary to evaluate the performance of dynamic olfactometry, especially for measuring pig house odour, and to investigate measures to optimise this method.

CHAPTER 2

RESEARCH OBJECTIVES AND THESIS OUTLINE

CHAPTER 2: RESEARCH OBJECTIVES AND THESIS OUTLINE

2.1 Research objectives

The overall objective of this thesis was to evaluate dynamic olfactometry, according to the CEN (2003)-standard, specifically for measuring pig house odour concentrations and to investigate measures to improve assessments by this method.

The specific objectives of this thesis therefore were:

- To critically review the bottlenecks of CEN (2003) for pig house odour measurements
- To get insight into the sources of variation when applying dynamic olfactometry
- To investigate the representativeness of the reference gas, n-butanol for pig house odour measurements
- To research measures on lab-scale to improve the precision of olfactometry

To enable these investigations, this PhD-study started with the build-up and development of an olfactometric laboratory at ILVO.

2.2 Thesis outline

A critical literature review was performed on the application of dynamic olfactometry for the measurement of livestock odours. This literature review is presented in Chapter 1.8.

At the start of this research, an olfactometric laboratory was built at ILVO, specifically dedicated to the measurement of pig house odour. The results of the odour panel selection and verification tests and of a first exploratory sampling campaign in a pig house are presented in chapter 3, as well as the results of a first participation in inter-laboratory comparison tests.

Specific methodological issues of CEN (2003) were further investigated by experiments and simulations. Here the focus was on the uncertainty related to olfactometric

measurements and the representativeness of the reference gas (n-butanol) for environmental odours, such as pig house odour (Chapter 4 & 5).

In chapter 4, an analysis of the sources of variation, when performing olfactometric measurements of both n-butanol and pig house odour are presented. The predictability of the n-butanol sensitivity of panellists for their pig house odour sensitivity was studied along with the transferability of the inter- and intra-panellist repeatability for n-butanol and pig house odour.

In chapter 5, the effect of different measures to improve the precision of olfactometric measurements is investigated, both for n-butanol and pig house odour. Also the transferability of the precision for n-butanol to pig odour was studied.

Chapter 6 discusses the implications of the research findings from chapters 3 to 5 for further practice, considering for example, efficiency assessments of odour abatement techniques. The main results of this PhD-study and some recommendations for future research are presented in chapter 7.

Figure 8 illustrates the specific subjects of interest of this thesis and the analysed variables.

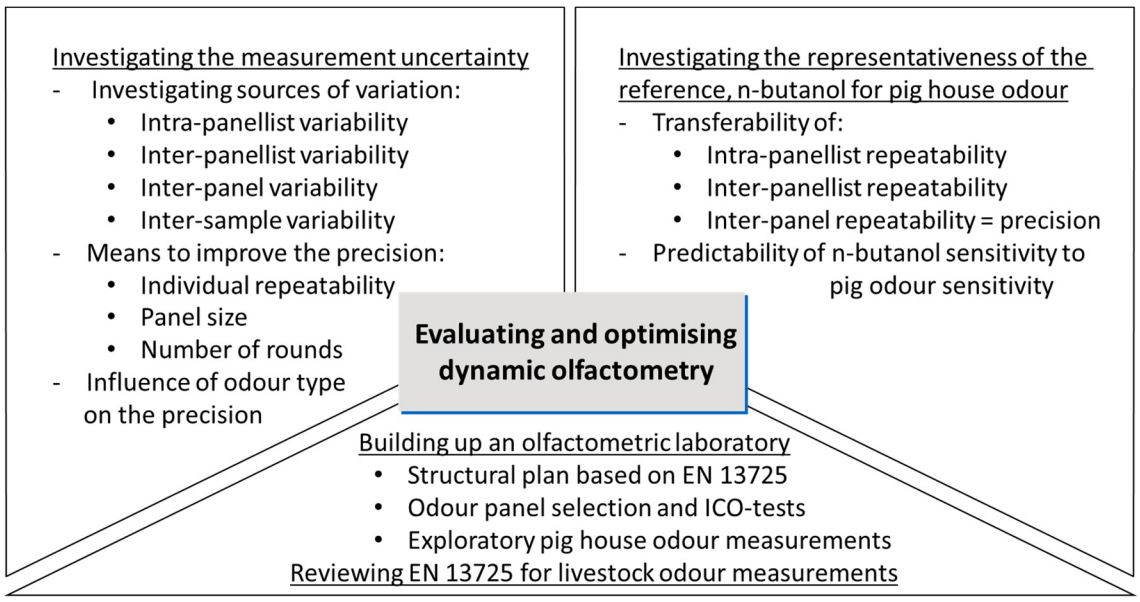


Figure 2.1 Thesis' subjects

CHAPTER 3

BUILD-UP OF AN OLFACTOMETRIC LABORATORY FOR THE MEASUREMENT OF PIG HOUSE ODOUR

CHAPTER 3: BUILD-UP OF AN OLFACTOMETRIC LABORATORY FOR THE MEASUREMENT OF PIG HOUSE ODOUR

The measurements and investigations described in this thesis were possible after the build-up and development of an olfactometric laboratory at ILVO during this PhD-study. This chapter depicts firstly the construction of the olfactometric laboratory at ILVO, elaborating as well on the infrastructure and equipment as on the odour panel selection process. Secondly, the results of an exploratory odour measurement campaign at a fattening pig farm in Diksmuide are presented. Thirdly, the results of the participation in interlaboratory comparison tests with environmental odours and n-butanol are depicted.

3.1 Build-up of the odour lab

An olfactometric odour laboratory was constructed at ILVO and made operational according to the requirements of CEN (2003).

3.1.1 Infrastructure and equipment

A scientific literature study on the CEN (2003)-requirements was performed and experts in the field (odour experts, acclimatisation experts, an architect) were consulted in order to constitute a suitable building plan for the olfactometric laboratory. The odour laboratory was acclimatized to ensure that the temperature fluctuated less than 3 °C and that the air in the odour laboratory was continuously refreshed with odourless air during the measurements. The room was put in over-pressure to avoid other air influx. An active carbon vessel of 75 kg (Desotec NV mobile unit Aircon 200 M with Airpel 10-3 active carbon, Roeselare, Belgium) was foreseen along with a particulate filter to clean the air entering the room and to make it odourless.

A calibrated olfactometer (Odournet TO8-olfactometer, Kiel, Germany), manufactured following the requirements of CEN (2003) and with Yes/No-presentation mode, was bought after consulting odour experts and performing a study on the performance of olfactometers present on the market. A compressor (Oil-free Atlas Copco LF - compressor, Air Compact Belgium bvba, Gentbrugge, Belgium) was also acquired to provide the olfactometer with compressed, odourless air for diluting the odour samples and to be able to present blanks to the panellists. On the compressed air-line, an air

cooler and dryer were foreseen to prevent condensation of the compressed air that is guided towards the olfactometer, together with a particulate filter to remove particulate material from the compressed air line. An active carbon filter (Donaldson Ultrafilter DF0035 with an A0120-active carbon cartridge, Donaldson bvba, Leuven) was also installed to removed residual odours from the compressed air. The incorporated fine particle filter removed activated carbon particles.

The certified reference material consisted of a pressurized gas cylinder of n-butanol diluted in nitrogen (59.22 ppmv n-butanol, with a spectroscopic quality of 99.9 % and expanded uncertainty of at most 5 %, which was certified by Westfalen AG, Münster, Germany).

The build-up of the odour laboratory implied investment costs (Table 3.1). Hereby it is assumed that a room for the laboratory is already available.

Table 3.1 Investment costs for an odour laboratory

Subject of investment	Costs (Euro)
For a suitable and air-conditioned room	30000
Equipment to generate odor-free compressed air	8000
Olfactometer (TO8, Yes/No)	60000

The odour laboratory was ready for use after about a year (end of 2011) (Fig. 3.1).

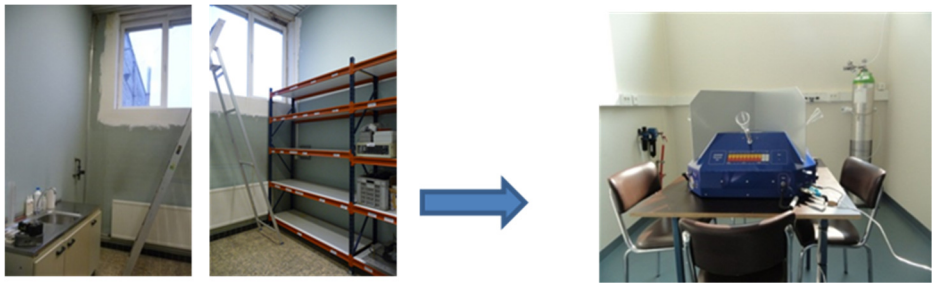


Figure 3.1 Building up an odour laboratory

Odour sampling equipment was made (Fig 3.2). The sampling bags were made from polyethyleneterephthalate (Nalophan film, Foodpack Benelux, Harderwijk, Netherlands) and polytetrafluoroethylene (PTFE) tubing (Teflon tubing, De Mulder NV, Ghent, Belgium) and corks (Teflon corks, Odournet, Kiel, Germany). These materials are allowed to come into contact with the odour sample according to CEN (2003). The tubing for sampling is also from PTFE.

The sampling cylinder was air-tight. The sampling pump was fixed to the outside of the sampling cylinder (Figure 3.2) and outside air could be sucked in the sampling bag

through the 'lung principle' (Chapter 1.6.1). In that way, the odour sample only came into contact with the sampling bag and tubing, which were were made of material that was suitable to come into contact with the odour sample according to CEN (2003).


(a)

(b)

Figure 3.2 Sampling apparatus: (a) with 10L-bag (b) with 40 L-bag

When building up an odour laboratory, re-occuring costs should also be considered (Table 3.2). The costs related to the sampling equipment are given in table 3.3.

Table 3.2 Re-occuring costs related to an odour laboratory

Re-occuring costs	Minimum costs (Euro)	Frequency
Calibration of olfactometer	1990	1x / year
Participation in interlaboratory comparison test	2000	1x / year
Certified n-butanol	552	1x / 2 year
Re-activation active carbon	615	1x / year
Active carbon element	142	Every 2/3 months
Maintenance compressor	147	< Working hours

Table 3.3 Costs related to the sampling equipment

Sampling equipment	Minimum costs (Euro)
1 Large sampling cylinder	502
1 Sampling pump	1732
1000 m Nalophane, 300 mm diameter => +/- 700 bags of 50 L	644
Teflon tubing incl. import cost (200 m, 6x8 mm)	1035
Teflon corks (20 pieces)	65

3.1.2 Panel selection process

In order to gain a qualified odour panel, multiple panellists' selection and verification tests were organised. Colleagues were invited to participate in the odour panel selection tests during presentations about the purpose of the odour lab, organised at the different ILVO-units (TV115, L&M, Plant 21, Directie, TV370, Plant 96-109). In total, 80 persons (including external relations of ILVO) participated voluntary in the panel selection tests.

From 2011 to 2014, 80 n-butanol measuring sessions were organized to test the suitability of the candidates for the odour panel and to evaluate their performance once selected, keeping a measuring history of each panellist according to CEN (2003).

To test whether a candidate was qualified for the odour panel, his sense of smell was tested (Fig 3.3) in at least 3 measuring sessions, performed on non-consecutive days.

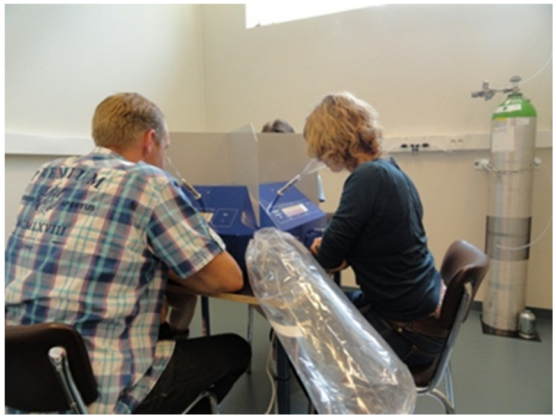


Figure 3.3 Panel selection test with n-butanol

At least 10 individual n-butanol threshold estimates were determined over these 3 sessions to verify the performance of the candidate's sense of smell. To qualify for the odour panel, a candidate's n-butanol results had to fulfil 2 criteria of CEN (2003) (Fig 3.4), namely:

- Selection criterion 1: individual repeatability criterion: The antilog of the standard deviation ($10^{s_{ITE}}$), calculated from the logarithms ($\log_{10}$) of the candidate's individual threshold estimates, expressed in mass concentration units of the reference gas, should be at most 2.3.
- Selection criterion 2: individual sensitivity criterion: The geometric mean of the individual threshold estimates ($\bar{y}_{ITE}$), expressed in mass concentration units of the reference gas, had to lie within the range of 20 to 80 ppb of n-butanol.

The performance of the qualified panellists had to be checked regularly, keeping a measuring history, as set forth by CEN (2003). During these follow-up measurements the panel selection criteria of the respective panellists were recalculated according to CEN (2003). The results from the panellists' selection and performance follow-up measurements can be found in Fig. 3.4, representing firstly the values for panel selection criterion 1 (individual repeatability) and secondly those for panel selection criterion 2 (individual sensitivity) of the 80 candidates. 32 of the 80 tested candidates passed both panel selection criteria. This comes down to 40 % of the tested candidates. Of the 48 candidates whom did not pass the panel selection procedure, 19 passed only for selection criterion 1, 14 passed only for selection criterion 2 and 15 of them did not pass for any of the two selection criteria. So, both selection criteria proved to be bottlenecks during panellists' selection. Amongst the qualified panellists were also external relations of ILVO which could not be employed on a regular basis. Considering the high drop-out of candidates through panel selection, it is clear that disposing of a sufficiently large odour panel for performing regular odour measurements, requires numerous panellists' selection tests.

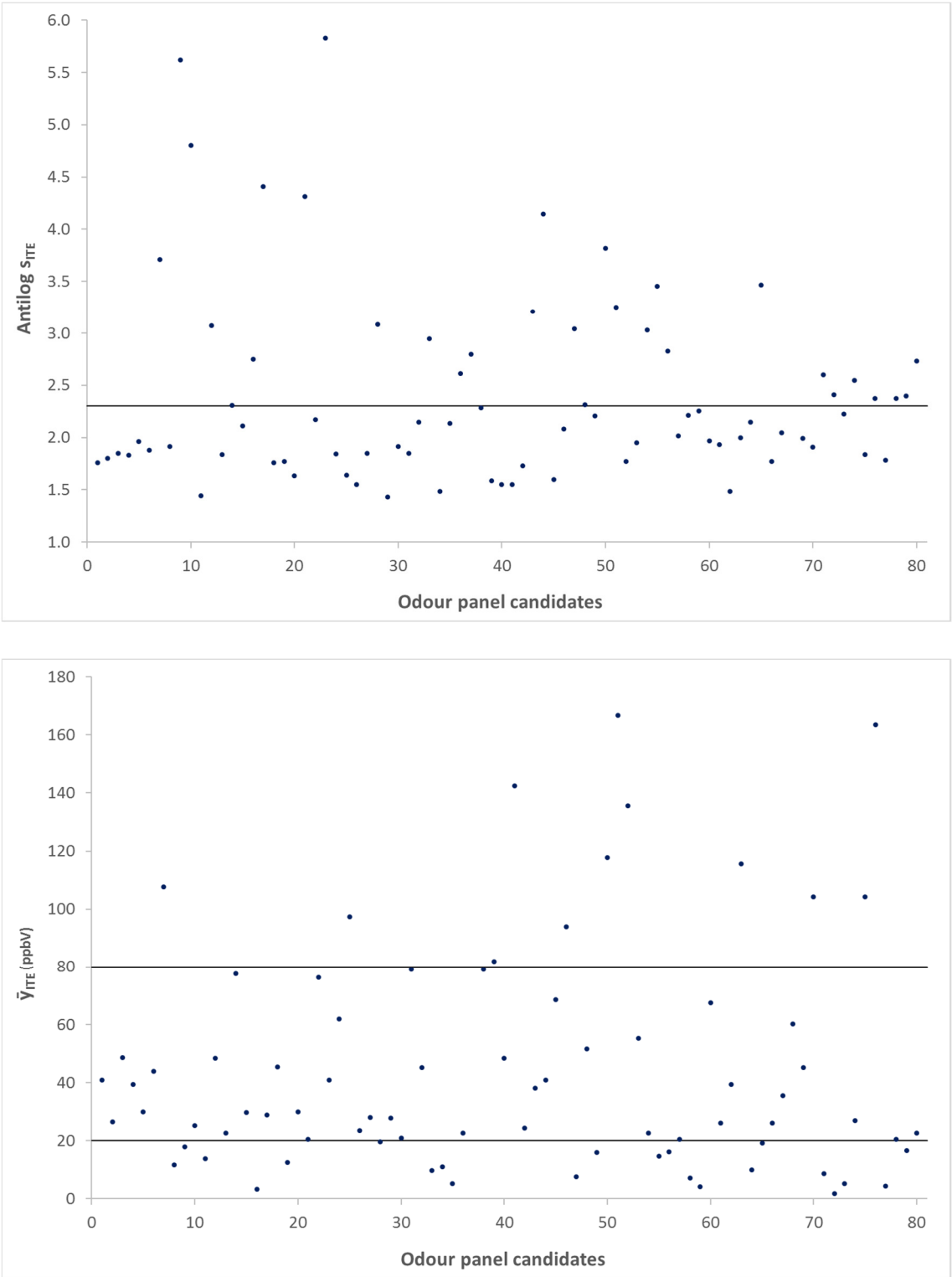


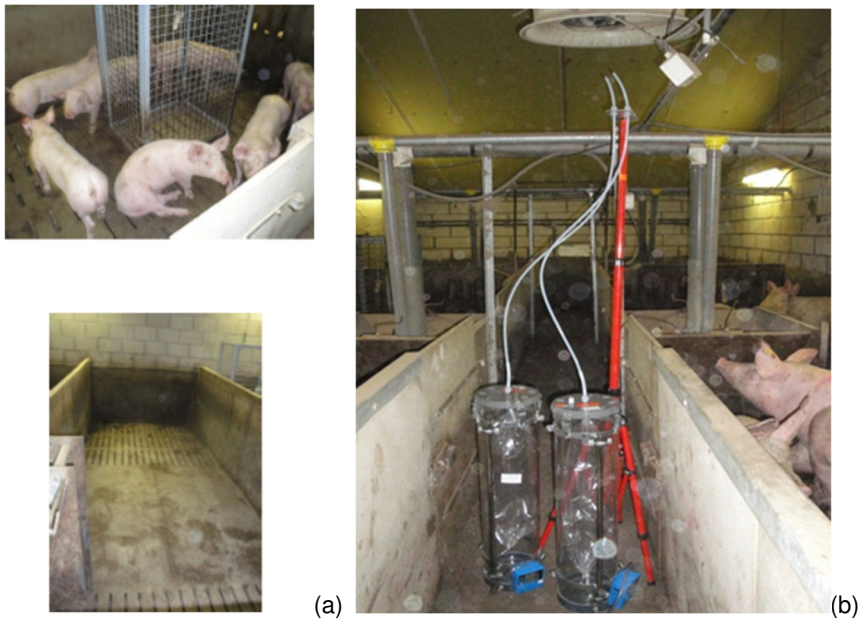
Figure 3.4 Panel selection results of the 80 candidates, considering (a) panel selection criterion 1, with indication of the CEN (2003) – limit value; (b) panel selection criterion 2, with indication of the CEN (2003) – upper and lower limit value

* The result of candidate 37 for panel selection criterion 2 was 610 ppb. The maximum represented value in the ordinate (y-axis) was limited to 180 ppb to enable a clear representation of the other 79 candidates.

** Candidate 68: antilog = 10.0

3.2 Exploratory sampling campaign

An exploratory pig odour sampling and measurement campaign was organized in the beginning of 2012 with the firstly selected panellists and candidates showing good performance in their first n-butanol tests. The purpose of this exploratory campaign was to gain experience in sampling and in measuring pig house odour. Its' purpose was also to get a first idea of typical indoor concentrations in pig houses. Odour samples then were taken at a pig fattening farm in Diksmuide (Hove et al., 2012). The campaign consisted of 12 sampling and accordingly 12 measuring days from January 2012 till June 2012. In total, 93 odour samples were taken at the pig farm in this period. The pig fattening farm had traditional and low ammonia emission compartments. The traditional compartments had fully slatted floors and the low ammonia emission compartments had sloped walls in the manure pit to reduce the emission surface (Hove et al., 2012, Fig 3.5a). All compartments were mechanically ventilated by door-roof ventilation, with separate ventilation fans per compartment. Odour samples were taken in duplicate (Fig 3.5b) inside two traditional and two ammonia emission low compartments. The duplicate samples were aspired simultaneously per compartment. Each sample was collected in a 10 L Nalophane bag, placed within a sampling cylinder, using the lung principle (CEN, 2003, Fig 12b). Sampling was done along a Teflon tube of 1 meter 30, which was flushed before sampling with compartment air. The sampling height was 20 cm beneath the ventilation duct. The sampling duration was 15 minutes per bag. The door of the respective pig house compartment was closed during sampling.



(a) (b)

Figure 3.5 Compartment floors and odour sampling at the fattening pig farm in Diksmuide

The odour samples were taken between 9:20 AM and 13:20 PM (Hove et al., 2012). Between January 16th and January 23rd 2012, one traditional and one ammonia low emission compartment were intensively cleaned with water and disinfectant (Hove et al., 2012). Before cleaning, these sampled traditional and low ammonia emission compartments contained 52 and 67 fattening pigs respectively (Hove et al., 2012). After the cleaning, these were filled with 59 and 69 fattening pigs, respectively. The other sampled traditional and low ammonia emission compartments were only cleaned dryly (removing manure) on the 23rd of December 2011 and they contained 60 and respectively 69 fattening pigs at the first sampling day. The pig house odour samples were measured with the panellists in the olfactometric within 30h after sampling, according to CEN (2003). 23 measuring sessions (each 90 min.) were organized over the 12 measuring days. One n-butanol sample and 4 pig house odour samples were measured per measuring session and this by a panel of 4 panellists following the CEN (2003)-procedure. Exceptionally, namely for 2 of the 23 measuring sessions, only 3 panellists were available at the start of the session. The simultaneously sampled pig odour samples were measured by the same odour panel. The n-butanol sample was measured in one or two rounds and each pig odour sample was measured in three to four rounds of dilutions. The range of the odour concentrations in the traditional and low ammonia emission compartments during the sampling period are given in table 3.4. It has to be mentioned that for 63 of the 89 pig odour samples measured with 4 panellists, the remaining panel size after retrospective screening was smaller than 4. Therefore, the results before retrospective screening, taking into account the thresholds of all 4 participating panellists, are also displayed in table 3.4. The data measured were in the same range of those determined in 2002 by Van Langenhove and Defoer (Van Langenhove & Defoer, 2002). The effect of type of compartment (traditional versus ammonia low emission) and type of cleaning (dry versus wet) (fixed categorical independent variables) on the odour concentrations (dependent variable) was tested with a mixed regression model with compartment as random factor to correct for repeated measurements within each compartment. The number of the fattening round, the day in that fattening round (as well as the interaction between day in round and round number) and the amount of fatting pigs present in the stable, were added as covariates.

The mixed regression model was built as follows:

$$Ypig_{ij} = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_4 + \beta_5 X_6 + \mu_j + e_{ij} \quad \text{(Eq 3.1)}$$

$Ypig_{ij}$ = pig house odour concentration [ou_E/m^3]

β_0 = intercept

β_1, = regression coefficient for the different independent variables

X_1 = cleaning effect (fixed effect) with X_1=0 for dry cleaning; X_1=1 for wet cleaning)

X_2 = compartment type (fixed effect) with X_2=0 for traditional; X_2=1 for ammonia emission low)

X_3 = the number of the fattening round with X_3=0 for the 2nd round; X_3=1 for the 3rd round)

X_4 = the day in that fattening round (continuous variable)

X_5 = the amount of fatting pigs present in the stable (continuous variable)

j= compartment and i= measurement

μ= random fault on compartment level

e= residual

None of these factors (X_3, X_4 and X_5) were found to have a significant effect and thus they were removed from the model. The univariable effects of X_1 and X_2 were further tested. The measurement results showed no significant effect of the wet and the dry cleaning procedure as well as no significant difference in odour concentrations between the traditional and low ammonia emission compartments. This is in line with the results from the parallel measurements of NH_3, CO_2, CH_4 and N_2O (Ulens, 2015), were also no significant effects were seen of compartment type and the cleaning procedure.

Table 3.4 Range of odour concentrations in the traditional and low ammonia emission compartments

Housing type	Measurement stage	Odour concentration range [ou_E/m^3]		
		Minimum	***Maximum***	***Geometric mean***
Traditional	Before retrospective screening	609	26008	2701
	After retrospective screening	683	26008	2223
Low ammonia emission	Before retrospective screening	575	26386	3791
	After retrospective screening	483	55109	3391

This exploratory measuring campaign initiated us to further perform panellists' selection and verification tests, enabling odour measurements with larger panel sizes (Chapter 4 and 5). The first results suggested differences in performance of panellists for n-butanol and pig odour. It appeared that the variation between ITE gathered in different rounds by individual panellists was larger for n-butanol than for pig odour. Next to that, it seemed that the variation between panel thresholds measured based on different rounds was larger for pig house odour than for n-butanol. These indications however required further investigations, as described in Chapter 4 & 5.

3.3 Participation in inter-laboratory comparison tests

ILVO's odour laboratory participated in interlaboratory comparison (ICO) - tests for environmental odours and for n-butanol in June 2013.

3.3.1 ICO-test environmental odours

For this ICO-test, *Olfascan NV* provided two odour samples taken after a biofilter and two odour samples taken within a pig house. 7 laboratories received replicates of these sources. The replicates were taken simultaneously (at the same time and sampling location) and were transported in the same way to the different laboratories and allowing an equal time span in between sampling and analysis in these laboratories. On the 11th of June 2013, the four odour samples were measured at ILVO by a panel of 4 panellists. A 5th panellist was foreseen as a back-up panellist. ILVO's results (Table 3.5), compared to the results of the other participating laboratories, are represented in figure 3.6. As there is no reference value available to assess the accuracy of these environmental odour samples, EN13725 (§ 5.3.3.3) considers that the best estimate of the reference value is given by the geometric mean of the results of all laboratories participating in the round robin test. The geometric mean of the results of all labs (Table 3.6) for the same odour source is represented in figure 3.6 by a red square. The limits of the 95% confidence interval (C.I.) (Table 3.6) on the results of all labs for the same odour source are indicated by orange squares (Fig 3.6). The results of our laboratory are indicated in black in figure 3.6. Our results for both odour sources (Table 3.5, Fig 3.6) were within the limits of the 95% C.I. (Table 3.6, Fig 3.6). Our results of the samples taken after the biofilter, approximated the geometric mean of all labs (Tables 3.5 and 3.6, Fig 3.6) and were for the pig house samples close to the geometric mean of all labs. These results could suggest a good accuracy for environmental odours. Considering the allowed repeatability factor of 3.0 between consecutive repetitions of the same sample, according to CEN (2003), and the measured factors of 1.1 and 1.6 between the replicate samples, our results could suggest also an CEN (2003)-fulfilling precision, considering these environmental samples.

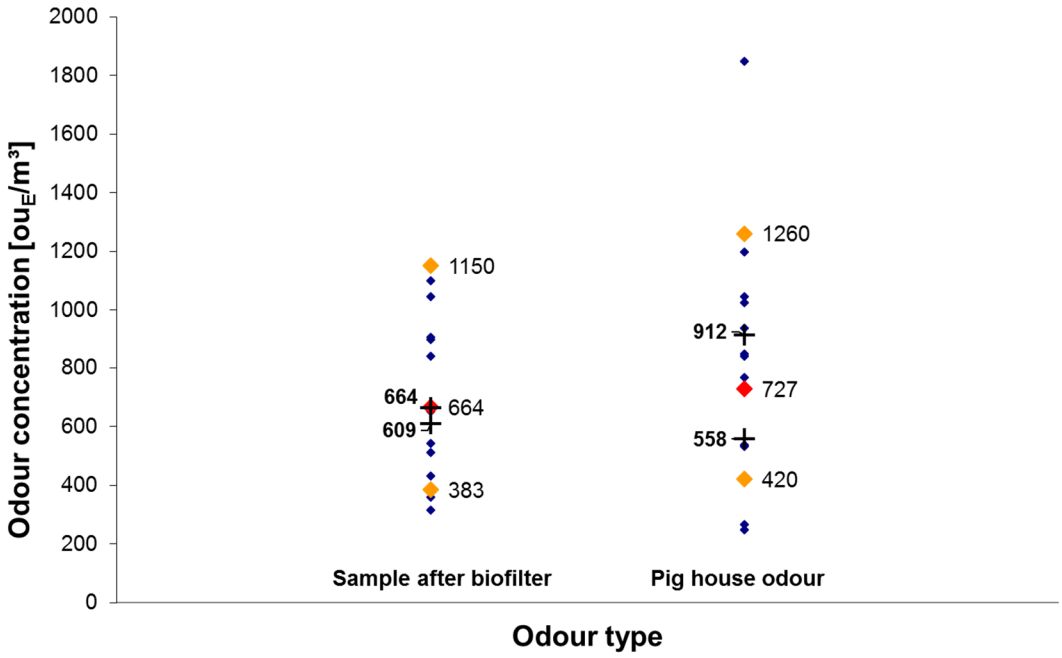


Figure 3.6 Results from the ICO-test on environmental odours (11/06/2013) (Van Elst, 2013). The results of ILVO are indicated (+). The 95 % confidence interval (◆) and geometric average of all laboratories (◆) for both odour samples are also given.

Table 3.5 Odour concentrations measured by ILVO during the ICO-test

Source	Replicate	Odour concentration [ou_E/m^3]
Sample after biofilter	1	609
	2	664
Pig house sample	1	558
	2	912

Table 3.6 Main results of the ICO-test conducted with 7 laboratories

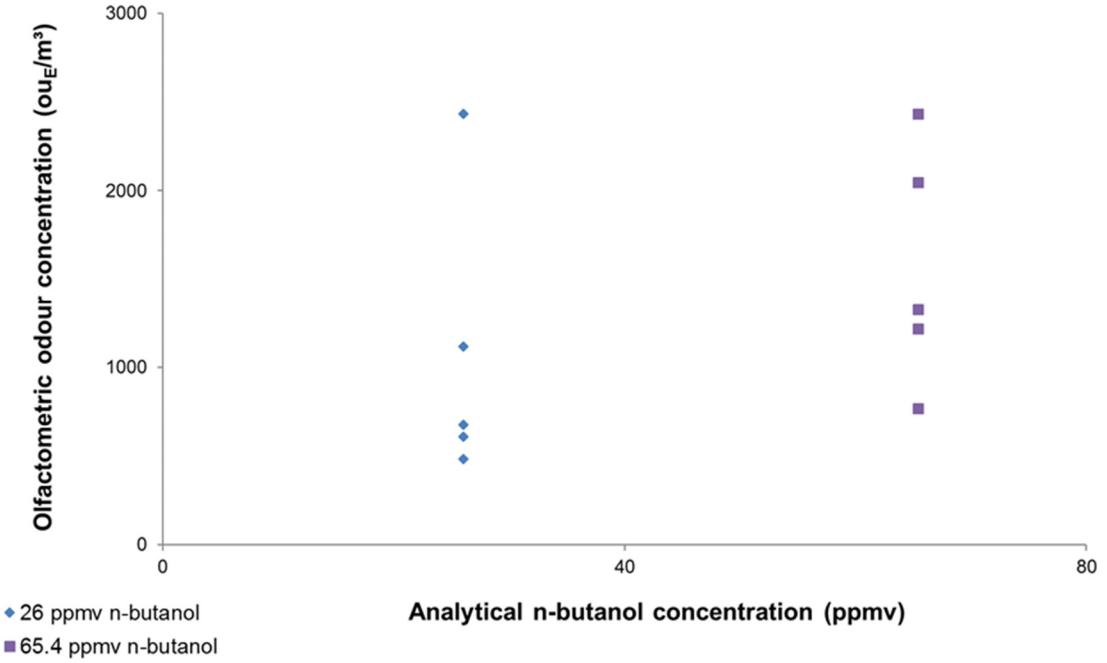
Source	N	Conc. range [$ou_E m^{-3}$]	Average conc. [$ou_E m^{-3}$]	95 % C.I. [$ou_E m^{-3}$]
Sample after biofilter	14	[315; 1163]	664	[383; 1150]
Pig house sample	14	[248; 1849]	727	[420; 1260]

3.3.2 ICO-test n-butanol

For the n-butanol ICO-test, ten 10 L gas cylinders were provided by *Odournet Gmbh*. The n-butanol concentration of these gas cylinders was certified by *Westfalen Gas Gmbh*. The odour concentrations of these 10 gas cylinders were measured by ILVO over two measuring days (24-25 June 2013). Before each measurement, a nalophane bag was filled from the respective gas cylinder. The odour concentration of each gas bottle was determined by olfactometry following the CEN (2003)-procedure and using at least four panel members. The results of the ICO-test, provided by *Odournet Gmbh*, showed that under the 10 unknown samples, two different analytical n-butanol concentrations were present, namely 26 and 65.4 ppmv n-butanol (Fig 3.7 a). The odour concentrations determined by ILVO's laboratory for the 10 odour samples and considering the two analytical concentrations of n-butanol, can be found in figure 3.7 a. The analytical n-butanol concentration [ppb] at which half of the panel could detect n-butanol (the panel threshold) during the ICO measurements is given in figure 3.7b. These analytical concentrations were calculated as the ratio between the analytical n-butanol concentration present in the gas bottle [ppbv] (communicated after the ICO-tests by *Odournet Gmbh*) and the odour concentration determined by the olfactometric measurement [$ou_E\ m^{-3}$]. It appeared that for 8 of the 10 samples the panel detected n-butanol at an analytical concentration between 23 and 54 ppbv (Fig 3.7b). These analytical concentrations are in the range of concentrations allowed for panellists' qualification according to selection criterion 2 (20-80 ppb) (Fig 3.7b). They are also close to the ideal value of 40 ppb (CEN, 2003) (Fig 3.7b). 2 of the 10 samples corresponded with an analytical concentration outside the range for panellists' selection criterion 2, namely 11 and 85 ppb. Based on these results, the accuracy limit of CEN (2003) was just fulfilled (A = 0.217), but the precision limit was not fulfilled.

The results of these exploratory odour measurements of n-butanol and environmental odours, together with the findings of the literature review, prompted us:

- To further investigate the transferability and predictability assumption of CEN (2003) for pig house odour (Chapter 4)
- To study the effect of selected precision improving measures (Chapter 5)



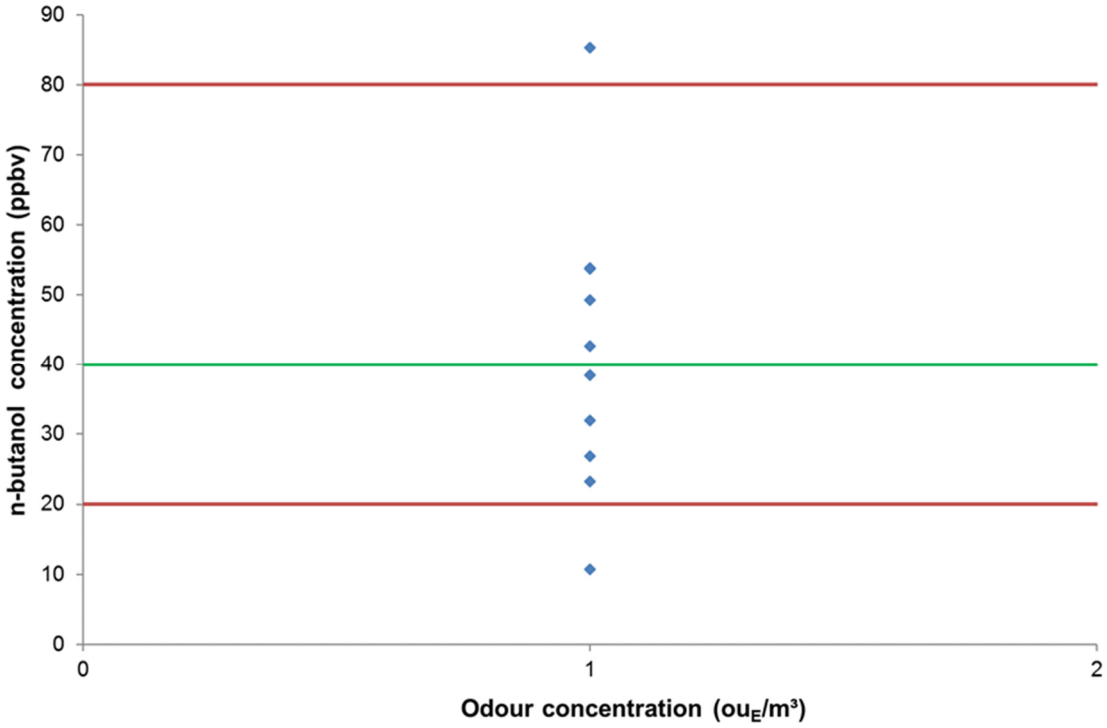


Figure 3.7 Results from the ICO-test on n-butanol (25-26/06/2013): a) Odour concentrations measured by dynamic olfactometry as a function of the analytical concentration of the n-butanol samples; b) Analytical concentration at the panel threshold according to these olfactometry measurements.

CHAPTER 4

COMPARATIVE ODOUR MEASUREMENTS ACCORDING TO EN 13725 USING PIG HOUSE ODOUR AND N-BUTANOL REFERENCE GAS

Redrafted from:

Hove, N. C. Y., Van Langenhove, H., Van Weyenberg, S., & Demeyer, P. (2016). Comparative odour measurements according to EN 13725 using pig house odour and n-butanol reference gas. *Biosystems Engineering*, 143, 119-127.

CHAPTER 4: COMPARATIVE ODOUR MEASUREMENTS ACCORDING TO EN 13725 USING PIG HOUSE ODOUR AND N-BUTANOL REFERENCE GAS

From the literature review (Chapter 1), it could be deduced that the representativeness of the reference gas, n-butanol, is being questioned by practitioners of dynamic olfactometry. During the exploratory odour measurements (Chapter 3), differences in performance of the odour panels for n-butanol and pig odour were noticed, but required a more profound investigation to assess their magnitude. In this chapter, the assumption of CEN (2003) regarding the predictability of the panellists' sensitivity (§3.3 in CEN (2003)) and the transferability of their repeatability (§5.1 in CEN (2003)) will be thoroughly investigated.

4.1 Introduction

Experts agree that when olfactometry is applied in practice a high amount of uncertainty is related to the measurements (Boeker et al., 2008; Boeker, 2014; Zarra et al. 2008; Zarra et al., 2009; Andersen, 2013) and that an estimation of the measurement uncertainty is vital for a correct interpretation of the results (Higuchi, 2009) by scientists and policy makers (Ubeda, Lopez-Jimenez, Nicolas, Calvet, 2013). According to Boeker et al. (2008) and Zarra et al. (2008), the scatter on olfactometric results is caused by the variability of the biological sense of smell. The large uncertainty results in a low reproducibility between laboratories. Therefore, efforts are necessary to reduce the measurement uncertainty (Clanton et al., 1999), but information on the most important source of variation is limited (Van Harreveld & Heeres, 1997; Clanton et al., 1999; Klarenbeek et al., 2014). Van Harreveld and Heeres (1997) reported that the variability in responses within and between panellists are a key source of variability in olfactometric results. According to the variability study by Clanton et al. (1999), inter- and intra-panellist variations were the most important sources of variation, while in the study by Klarenbeek et al. (2014), the laboratory seemed the most important compound of variance for n-butanol measurements and for non-butanol odorants this seemed to be the panel.
As explained in section 1.8.1, the substance n-butanol is used as a reference material and for panel selection purposes, based on the assumption of CEN (2003) that the

panellists' sensivity to n-butanol will be a predictor for their sensitivity to other environmental odours (§3.3 in CEN (2003)) and specifically that the performance characteristics (in terms of accuracy and precision) of qualified assessors for n-butanol are transferable to other environmental odours (§5.1. in CEN (2003)). Currently, however questions are rising regarding the representativeness of n-butanol for measuring agricultural odours (Laor et al., 2014; Parker et al., 2005) and more generally for environmental odours (Klarenbeek et al. 2014) as indicated in section 1.8.1.

In summary, there is a need for a better insight into the factors that contribute to the variability in olfactometric results. It is also necessary to study the predictability of the n-butanol sensitivity of qualified panellists for pig house odour. The transferability of the repeatability of panellists for n-butanol towards pig house odour should be investigated. In this research, odour sampling and measurements were carried out with the aim of providing answers to the following questions:

- What is the most important source of variation in the olfactometric determination of n-butanol and pig house odour thresholds, when comparing the influence of samples, of panels, of panellists and of panellist's repetitions?
- Is the repeatability of panellists (within and between panellists) to n-butanol similar for pig house odour?
- Can the sensitivity of a panellist (i.e. detection threshold) to pig house odour be predicted by his/her sensitivity (detection threshold) to n-butanol?

4.2 Materials and methods

4.2.1 Sampling campaign

Pig house odour samples were taken at a practise farm on 7 sampling days during October and November 2014. On each day, 2 consecutive samplings were performed in duplicate. All samples were taken in the central hallway at approximately 1.6m height and between 2 p.m. and 5 p.m. Each sample of 40 l was collected in a Nalophan (Foodpack Benelux, Harderwijk, Netherlands) bag, which was placed within a sampling cylinder. The duplicate sampling bags were filled in 27 minutes using the lung principle (CEN, 2003) through 1 mutual sampling inlet (Fig. 4.1). Per sampling day, one of the two duplicate pairs was used for analysis (see section 2.3). The other duplicate pair served as backup samples.

Figure 4.1 Set-up for odour sampling in duplicate

During the first 6 sampling days, samples were taken at the same pig house equipped with an automatic ventilation control unit. The average ventilation rate (in m^3 h^{-1}) during sampling was obtained via readings from the Fancom control unit (De Jaeger, Aalter, Belgium): 13 to 24 measurements of the ventilation rate were noted during the sampling duration of 27 min. The last sampling was performed in another pig house without a ventilation control unit.

In the first pig house, experiments started with 126 fattening pigs of 23 weeks old. In the second pig house, 104 fattening pigs of 26 weeks old were present at sampling time.

The samples were transferred to the odour laboratory and stored there in their containers till the analysis was performed the following day. The temperature in the odour laboratory was regulated at a temperature similar to that present in the pig house during sampling (20.9 – 23.0 °C). No condensation occurred in the sampling bags during storage.

The n-butanol samples were sampled on the measuring days directly (i.e. undiluted) from a gas cylinder in the laboratory which contained a gas mixture of 61.1 ppmv n-butanol in nitrogen (calibrated and certified concentration by Westfalen AG, Münster, Germany).

4.2.2 Odour panel characteristics

The 19 panellists participating in the measurement campaign complied with both panel selection criteria of CEN (2003), based on their 10 most recent n-butanol thresholds. The average individual repeatability (x) of the panellists varied between 1.43 to 2.14 (CEN Crit. 1 ($x < 2.3$)). The average individual sensitivity (y) of the panellists varied between 20.4 ppb and 68.5 ppb (CEN Crit. 2 ($20 \leq y \leq 80$)). A panel size of 6 was preferred and all odour panels consisted of 5 to 6 qualified panellists.

4.2.3 Olfactometric analyses

On each measuring day 4 samples were analysed, which were 2 n-butanol samples and 2 in duplicate sampled pig house odour samples. The 4 samples were measured by two panels of 5 to 6 panellists on a 4-ports olfactometer with a 'Yes-No' presentation mode (Odournet TO8-olfactometer, Kiel, Germany) and this was done within three to four measuring sessions. The olfactometer was calibrated in August 2014. A measuring session lasted 1 hour and 10 minutes. Each odour sample was analysed by a panel in 3 ascending dilution series. The 4 samples were measured during a measuring session in the following order: first n-butanol, then pig house odour, then n-butanol and last the duplicate pig house odour sample. The measurements of the pig house odour samples started at around 16 to 18 hours after the sampling. The pig house odour samples were fully measured by the two panels within at least 20 hours to at most 26 hours after the sampling. The composition of the panels that measured the same samples within one measuring day were completely different, so 11 to 12 different persons participated per measuring day. The composition of the panels did also differ for the different measuring days: over the 7 measuring days, the panels had a different composition of 5 to 6 panellists out of the 19 qualified panellists.

The panellist's results for the 2 n-butanol samples were considered as repetitions of one n-butanol sample (§4.2.4.2), as both n-butanol bags were filled from one gas bottle with a fixed concentration. The panellist's results for the simultaneously sampled pig house odour samples were also considered as repetitions of the respective pig house odour of that sampling day, as pig house air for both bags was aspired through one common inlet at the same place, at the same time and with the same sampling velocity (Fig. 4.1).

4.2.4 Data analyses

4.2.4.1 Determination of the odour concentration and emission

Per measuring day, the odour concentration of the 4 samples was calculated per panel (of 5 to 6 panellists), based on the individual odour detection thresholds of the participating panellists. The nominal value of the odour concentration of a sample in ou_E m^{-3} was calculated as the geometric mean of the individual threshold estimates of the participating panellists, after retrospective screening (CEN, 2003). Retrospective screening (r.s.) was applied according to the procedure described in CEN (2003). Retrospective screening (CEN, 2003) aims at detecting exceptional deviant responses of qualified panellists, which could be caused by physiological factors (e.g. temporarily decreased or increased odour sensitivity induced by nasal congestion or irritation; specific lack of sensitivity or extreme sensitivity for an odour component of the analysed sample) or by psychological factors, such as decreased attention. The results of a panellist showing deviant sensory responses for a sample are excluded from the calculation of the odour concentration of that sample, as required by CEN (2003). A theoretical illustration of the retrospective screening procedure according to CEN (2003) can be found in the appendix A and a further description in Chapter 5.2.2.

The pig house odour emissions during sampling on the first 6 sampling days could be estimated by multiplying the odour concentration of the respective sample with the average ventilation rate during sampling.

4.2.4.2 Statistical analyses

All statistical analysis was done on the individual n-butanol and pig house odour thresholds of the panellists after r.s.. This means that per panellist, the logarithm of the geometric mean (log mean) of his/her 3 odour thresholds for each sample was calculated and used in the statistical analyses. The dataset for pig house odour was set up according to the following structure: on each measuring day one sample was measured by two panels of up to 6 panellists. Each panellist repeated the measurement of the considered sample twice through the measurements of the duplicate samples (section 4.2.3). The dataset for n-butanol was set up similarly, containing the following levels of data: sample, panel, panellist and repetition.

To investigate the most important source of variation in the determination of n-butanol and pig house odour thresholds, the variance components on the different levels of the

data were determined. Four-level (sample, panel, panellist, repetition) null-models (intercept only) were fit for individual n-butanol thresholds and for individual pig house odour thresholds as dependent variables using SAS 9.3 (Proc. Mixed, SAS Institute Inc., NC, USA). The model estimates the variation between different samples (variation at the sample level), the variation between the results of different panels for the same sample (variation at the panel level), the variation between the results of different panellists for the same sample (variation at the panellist level) and the variation between different repetitions of the same panellist (variation at the repetition level). Sample, panel and panellist were added as random factors to the model to correct for clustering of panels within samples, for clustering of panellists within panels and for repeated measurements within panellists, respectively.

Null-models (intercept only models):

$$Ybut_{ijkl} = \beta_0 + \mu_l + \mu_{kl} + \mu_{jkl} + e_{ijkl} \quad \text{(Eq 4.1)}$$

$$Ypig_{ijkl} = \beta_0 + \mu_l + \mu_{kl} + \mu_{jkl} + e_{ijkl} \quad \text{(Eq 4.2)}$$

Intraclass correlation at different levels (percentage variation at the different levels) ρ:

At sample level: $\rho_i = \sigma^2_{\mu l} / (\sigma^2_{\mu l} + \sigma^2_{\mu kl} + \sigma^2_{\mu jkl} + \sigma^2_{eijkl})*100$

At panel level: $\rho_i = \sigma^2_{\mu kl} / (\sigma^2_{\mu l} + \sigma^2_{\mu kl} + \sigma^2_{\mu jkl} + \sigma^2_{eijkl})*100$

At panellist level: $\rho_i = \sigma^2_{\mu jkl} / (\sigma^2_{\mu l} + \sigma^2_{\mu kl} + \sigma^2_{\mu jkl} + \sigma^2_{eijkl})*100$

At repetition level: $\rho_i = \sigma^2_{eijkl} / (\sigma^2_{\mu l} + \sigma^2_{\mu kl} + \sigma^2_{\mu jkl} + \sigma^2_{eijkl})*100$

$\sigma^2_{\mu l}$ = Variance of the sample level errors μ_l

$\sigma^2_{\mu kl}$ = Variance of the panel level errors μ_{kl}

$\sigma^2_{\mu jkl}$ = Variance of the panellist level errors μ_{jkl}

σ^2_{eijkl} = Variance of the repetition level errors e_{ijkl}

Second, the number of the repetition (1 versus 2) was added to the regression model as a fixed effect.

Models to test fixed effects:

$$Ybut_{ijkl} = \beta_0 + \beta_1 X + \mu_l + \mu_{kl} + \mu_{jkl} + e_{ijkl} \quad \text{(Eq 4.3)}$$

$$Ypig_{ijkl} = \beta_0 + \beta_1 X + \mu_l + \mu_{kl} + \mu_{jkl} + e_{ijkl} \quad \text{(Eq 4.4)}$$

$Ybut_{ijkl}$ = individual, measurement specific, n-butanol house odour thresholds

$Ypig_{ijkl}$ = individual, measurement specific, pig house odour thresholds

β_0 = intercept

β_1 = regression coefficient for X

X = the number of repetition (1 versus 2) (Fixed effect)

I = repetition, j = panellist, k = panel, l = sample

μ = random fault on different levels

e = residual

The intra-panellist repeatability (i.e. repeatability within panellists) was compared for n-butanol and for pig house odour. The repetitions made by panellists when evaluating the samples taken in duplicate were investigated. Coefficients of variation (C.V.s) were calculated on the repetitions made by individual panellists and this for n-butanol and for pig house odour, respectively. A non-parametric test (Wilcoxon test) was conducted to test the null-hypothesis that the median C.V. for n-butanol repetitions was not significantly different from the median C.V. for pig house odour repetitions. Bland-Altman plots (Dohoo et al., 2010) were made for both n-butanol and pig house odour to visualise the difference between two repetitions against their mean value, together with the 95% limits of agreement between repetitions and the mean difference between repetitions.

The inter-panellist repeatability (i.e. repeatability between panellists) during evaluation of n-butanol and pig house odour samples was compared. The coefficient of variation was calculated on the individual thresholds of each group of 11 to 12 panellists (2 panels) that measured the same sample. This was done separately for n-butanol samples and for pig house odour samples. A non-parametric test (Wilcoxon test) was used to test the null-hypothesis that the median C.V. of n-butanol samples and the median C.V. of pig house odour samples were not significantly different.

In order to study whether the n-butanol sensitivity of a panellist could predict his/her pig house odour sensitivity, a four level mixed regression model for individual pig house odour thresholds (dependent variable) as a function of individual n-butanol odour thresholds (fixed effect) was tested. Sample, panel and panellist were added as random factors. The contribution of the variation in n-butanol thresholds to the variation in pig house odour thresholds was also studied by comparing the variance components from the null-model for pig house odour with those from the final model for pig house odour as a function of n-butanol.

$$Ypig_{ijk} = \beta_0 + \beta_1 X + \mu_l + \mu_{kl} + \mu_{jkl} + e_{ijkl} \quad \text{(Eq 4.5)}$$

$Ypig_{ijk}$ = individual pig house odour thresholds

β_0 = intercept

β_1 = regression coefficient for X

X = individual n-butanol odour thresholds (fixed effect)

l = repetition, j = panellist, k = panel, l = sample

μ= random fault on different levels

e= residual

The contribution of the variation in n-butanol thresholds to the variation in pig house odour thresholds (explained variance with n-butanol) was also studied by comparing the variance components from the null-model for pig house odour with those from the final model for pig house odour as a function of n-butanol.

Explained variance with n-butanol:

At repetition level: $R^2_1 = (\sigma^2_{eijkl\ (Null)} - \sigma^2_{eijkl\ (Final)}) / \sigma^2_{eijkl\ (Null)} * 100$

At panellist level: $R^2_2 = (\sigma^2_{ejkl\ (Null)} - \sigma^2_{ejkl\ (Final)}) / \sigma^2_{ejkl\ (Null)} * 100$

At panel level: $R^2_3 = (\sigma^2_{\mu kl\ (Null)} - \sigma^2_{\mu kl\ (Final)} / \sigma^2_{\mu kl\ (Null)} * 100$

At sample level: $R^2_4 = (\sigma^2_{\mu l\ (Null)} - \sigma^2_{\mu l\ (Final)}) / \sigma^2_{\mu l\ (Null)} * 100$

$\sigma^2_{\mu l\ (Null)}$ = Variance of the sample level errors μ_l in the null model

$\sigma^2_{\mu kl(Null)}$ = Variance of the panel level errors μ_{kl} in the null model

$\sigma^2_{\mu jkl(Null)}$ = Variance of the panellist level errors μ_{jkl} in the null model

$\sigma^2_{eijkl\ (Null)}$ = Variance of the repetition level errors e_{ijkl} in the null model

$\sigma^2_{\mu l\ (Final)}$ = Variance of the sample level errors μ_l in the final model

$\sigma^2_{\mu kl(Final)}$ = Variance of the panel level errors μ_{kl} in the final model

$\sigma^2_{\mu jkl(Final)}$ = Variance of the panellist level errors μ_{jkl} in the final model

$\sigma^2_{eijkl\ (Final)}$ = Variance of the repetition level errors e_{ijkl} in the final model

All statistical tests were performed at a significance level of 0.05. All analyses were done using SAS 9.3. The models with fixed effects were called final models.

4.3 Results

4.3.1 Odour concentrations and emissions

Table 4.1 presents the average odour concentration ± standard deviation per odour type and the corresponding total number of measuring days, total number of panels and the respective number of odour concentration determinations.

The C.V. on the odour concentrations of the pig house odour samples of pig house 1 was 45 %. A C.V. of 45 % was also found on the odour concentrations of the corresponding n-butanol samples. This suggests that the odour concentration in pig house 1 did not change substantially during the sampling campaign as the variability for pig house odour is of the same magnitude as that for n-butanol. Through retrospective screening, for pig house odour, the thresholds of one panellist were excluded in 25 % of the panels, while for n-butanol this occurred in 43 % of the panels. Furthermore, for n-butanol the thresholds of 2 panellists were excluded from 7 % of the panels, while for pig house odour this never occurred.

Table 4.1 Average odour concentration of n-butanol and pig house odour ± standard deviation

Odour type	Sampling location	N days[1]	N panels[2]	N od. conc.[3]	Average odour concentration ± standard deviation (ou_E m^{-3})
n-butanol	Odour lab	7	14	28	1832 ± 819
pig house odour	Pig house 1	6	12	24	1839 ± 820
	Pig house 2	1	2	4	1808 ± 501

[1] Total number (N) of measuring days
[2] Total number (N) of panels
[3] Total number (N) of odour concentration determinations

The average (estimated) pig house odour emission was 28.7 (± 7.6) ou_E s^{-1} $animal^{-1}$. Therefore, this is in the same order of magnitude as the odour emission factors for fattening pigs in Flanders found by De Bruyn et al. (2001) (25.4 ou_E s^{-1} $animal^{-1}$) and Van Langenhove and Defoer (2002) (29.2 ou_E s^{-1} $animal^{-1}$) and of the emission factor reported in the Dutch regulation on odour nuisance in animal production (Ogink, 2010) (17.9 ou_E s^{-1} $animal^{-1}$). The C.V. over all odour emission estimations was 26 %. This relative variation in odour emissions (26 %) is smaller than the relative variation in odour concentrations (45 %). This can be attributed to the inverse relation between the odour concentration and the ventilation rate.

4.3.2 Sources of variation

The variance components, attributed to different sources of variation (sample, panel, panellist and repetition) in individual n-butanol thresholds and pig house odour thresholds are presented in Table 4.2.

The largest proportion of variation in n-butanol thresholds (68 %) and in pig house odour thresholds (58 %) is at the level of repetitions (measurement by the same panellist of the samples taken in duplicate) (Table 4.2). However, the effect of repetitions was found to be non-significant.

The second largest contribution to the total variation in pig house odour thresholds was situated at the sample level (32 %), whereas for n-butanol this was situated at the panellist level (Table 4.2). The smallest part of the total variation was at the panel level for both n-butanol and pig house odour (9 % and 2 %, respectively) (Table 4.2).

Table 4.2 Variance components at each level of the null-model for n-butanol and pig house odour. The largest proportion of variance is given in bold.

Type of odour	Source of variation	Variance components from the null-model	
		Var. Est.[1]	%[2]
n-butanol	Sample	0.011	10
	Panel	0.01	9
	Panellist	0.014	13
	Repetition	0.071	**68**
	Total Variance	0.105	100
pig house odour	Sample	0.026	32
	Panel	0.002	2
	Panellist	0.006	7
	Repetition	0.047	**58**
	Total Variance	0.082	100

[1] Variance estimate (Var. Est.).
[2] Proportion of variance present at different levels (= interclass correlation at different levels).

4.3.3 Repeatability of panellists for n-butanol and for pig house odour

4.3.3.1 Intra-panellist repeatability

Although the median C.V. was twice as high for n-butanol repetitions (4.4 %) as for pig house odour repetitions (2.3 %), no significant difference was found between their respective median C.V.

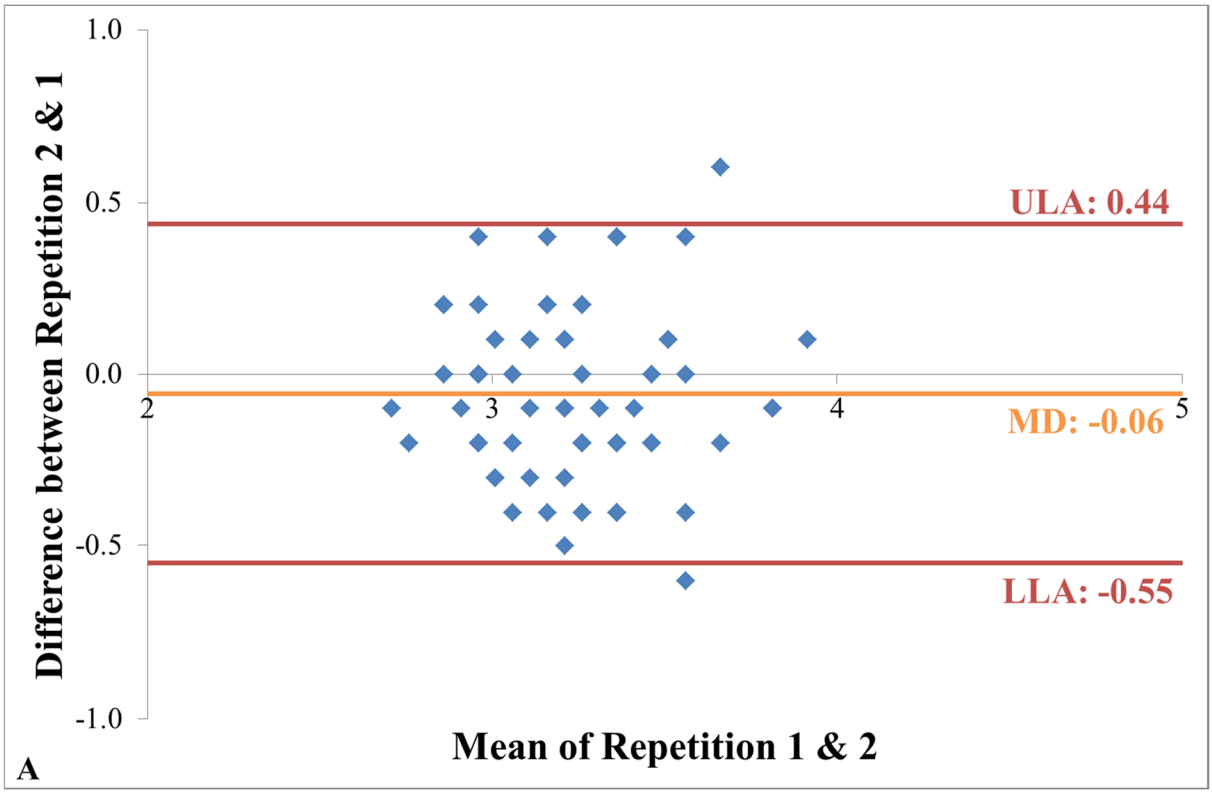
The Bland-Altman plots (Figure 4.2) illustrate that the confidence interval on the difference between repetitions of pig house odour (0.80) is somewhat smaller than that of n-butanol (0.99). This could imply that panellists are more repeatable for pig house odour than for n-butanol. However, the difference in the width of both these confidence intervals is very small. In this way, the Bland-Altman plots (Figure 4.2) visually confirm the result of the non-parametric test.

4.3.3.2 Inter-panellist repeatability

No significant difference was found between the median C.V. for n-butanol (8.6 %) and that of pig house odour (6.2 %), although it was larger for n-butanol.

4.3.4 Predictability of n-butanol sensitivity for pig house odour sensitivity

After running the null-model for pig house odour (see section 4.3.2), the individual n-butanol thresholds of panellists were added as a fixed effect to the model (final model). The individual n-butanol thresholds were found to have a significant effect on the individual pig house odour thresholds ($p=0.027$). The estimation of the slope of the regression was 0.15. The change in individual panellist thresholds for n-butanol is thus weakly related to the change in individual panellist thresholds for pig house odour (Fig. 4.3). Furthermore, the confidence interval on the estimation of the slope was very large ([0.02, 0.29]). Only a small part of the variation in pig house odour sensitivity could be explained by n-butanol sensitivity (Table 4.3).



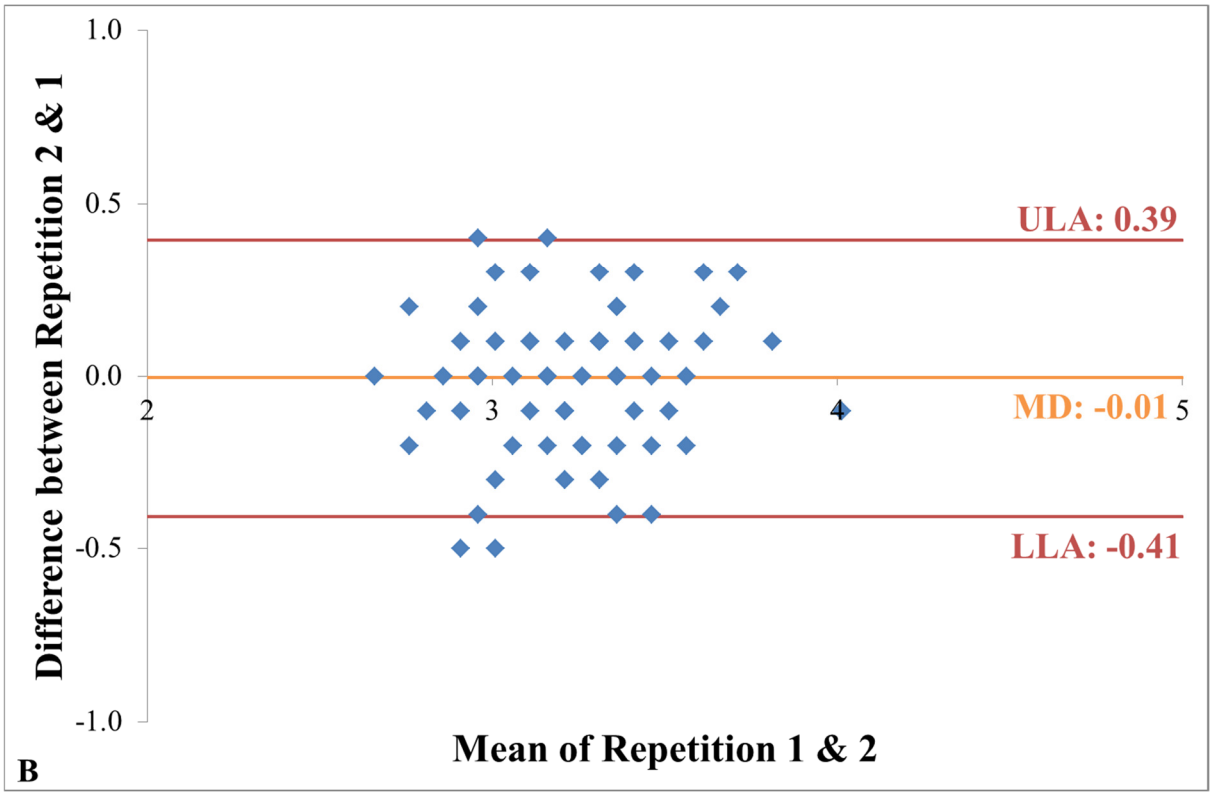


Figure 4.2 Bland Altman plot of n-butanol (A) and of pig house odour (B), with indication of the observed mean difference (MD) between repetitions and the 95 % upper and lower limit of agreement (ULA and LLA) for agreement between repetitions.

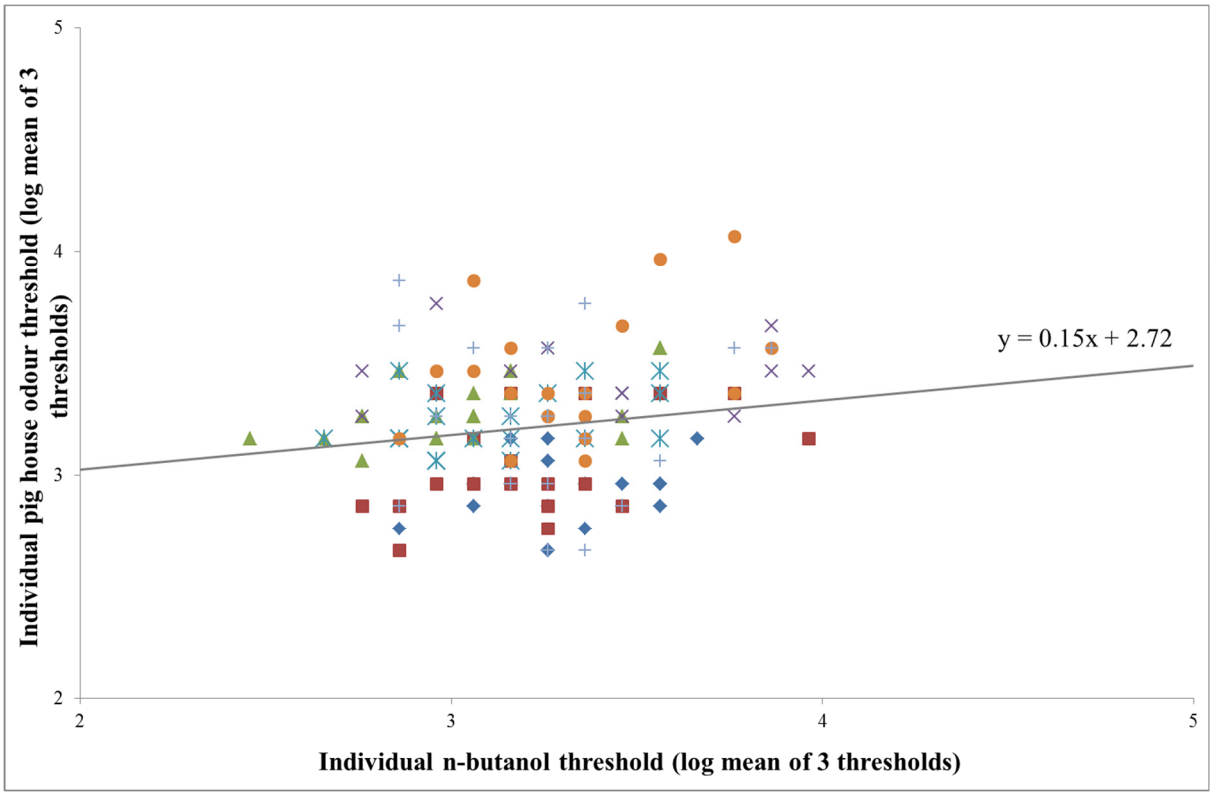


Figure 4.3 Individual pig house odour thresholds as a function of individual n-butanol thresholds for sample 1 (◆), sample 2 (■), sample 3 (▲), sample 4 (✖), sample 5 (✳), sample 6 (●), sample 7 (✚) and the resulting equation of the final linear regression model.

Table 4.3 Variance estimates at each level (Sample, Panel, Person, Repetition) of the null-model and the final model (n-butanol threshold as fixed effect) for pig house odour thresholds.

Source of variation	Variance estimates	
	Null-model	***Final model***
Sample	0.026	0.031
Panel	0.002	0.001
Panellist	0.006	0.005
Repetition	0.047	0.043
Total Variance	0.082	0.08

4.4 Discussion

4.4.1 Sources of variation

The most important source of variation in both n-butanol and pig house odour thresholds was the individual variability of panellists. Nevertheless, the variation due to repetitions was found statistically non-significant. This suggests that the overall variation in odour thresholds of both odours was small. The smallest variation (for both odour types) resided between panels, suggesting that our panels generated similar results and therefore were interchangeable. It could be expected that the variation due to the sample was more important for pig house odour than for n-butanol, since the pig house odour samples were taken under different sampling conditions (on different days), while all n-butanol samples analytically had the same concentration.

Clanton et al. (1999) followed a different approach in their variance study on odour providing from a swine facility. This study also dated prior to the establishment of the CEN (2003) standard (2003). Clanton et al. (1999) found that panel, panellist, day, time block within a session (beginning or middle or end) and odour sample strength had a significant effect on the odour concentration. The variation between panellists and within panellists were the largest sources of variation in this study and were comparable in magnitude. From our study the variation between panellists had a much smaller contribution (5 and 8 times smaller for n-butanol and pig house odour, respectively) than the variation within panellists (Table 4.2). The approach of the variance analysis by Klarenbeek et al. (2014) was also different. Klarenbeek et al. (2014) studied the following levels of variation: laboratory, panel, panel session and replicate measurement, on n-butanol and non-butanol odorants. The study was based on panel thresholds. The variation between panellists within a panel and the variation within panellists making repetitions, thus was not quantified in the study by Klarenbeek et al. (2014).

4.4.2 Repeatability of panellists for n-butanol and pig house odour

4.4.2.1 Intra-panellist repeatability

It appeared that the intra-panellist repeatability was better for pig house odour than for n-butanol (median $C.V._{\text{pig house odour}}$ < median $C.V._{\text{n-butanol}}$). This could be expected from the study by Laska and Hudson (1991), who found a clear trend towards lower variability within persons by increasing the odour complexity. However, no significant difference was found between the intra-panellist repeatability for n-butanol and for pig house odour

in this study. This is in agreement with the results of Van Harreveld and Heeres (1995), who found a lower spread on the ratios between panellists' individual thresholds and the panel's threshold for environmental odours ($s_{\Delta Z\_env}$=2.2) than for n-butanol ($s_{\Delta Z\_but}$=2.6), but the difference in spread was proven non-significant. Comparable findings were also reported by Defoer and Van Langenhove (2004), who compared individual variances of panellists (on the individual thresholds in two consecutive dilution series) for n-butanol and pig house odour. They found that the average individual variance (across multiple samples) and the standard deviation on the individual variances, were larger for n-butanol than for pig house odour. However, the differences were found to be non-significant. Our results can also be considered in line with McGinley and McGinley (2010) who found that CEN (2003)-qualified panellists also generated precise results for H_2S measurements (H_2S is an important component of pig house odour).

4.4.2.2 Inter-panellist repeatability

It seemed that the inter-panellist repeatability was better for pig house odour than for n-butanol (median C.V.$_{\text{pig house odour}}$ < median C.V.$_{\text{n-butanol}}$). From section 4.3.1, the exclusion of panellists through retrospective screening occurs less frequently when measuring pig house odour than when measuring n-butanol. Therefore, the responses of individual panel members within a panel were more alike when measuring pig house odour compared to n-butanol, again suggesting better inter-panellist repeatability for pig house odour than for n-butanol. This would be in the line with the results of the study by Laska and Hudson (1991) who found a lower variability in thresholds between panellists with increasing stimulus complexity. However, no significant difference was found between the inter-panellist repeatability for n-butanol and pig house odour. This is in accordance with the results of Van Harreveld and Heeres (1995) who found that the standard deviation on the ratios ($s_{\Delta Z}$) between panellists' individual thresholds and the panel's threshold for environmental odours was not significantly different than that for n-butanol.

4.4.3 Predictability of n-butanol sensitivity for pig house odour sensitivity

The results of the present study, showing a weak relation between individual panellist's thresholds for n-butanol and for pig house odour, could not confirm that individual n-butanol thresholds are good predictors for individual pig house odour thresholds. These results agree with the results of Defoer and Van Langenhove (2004) considering their complete dataset. They found that variances on pig odour ratios and on n-butanol ratios

(see annexe) were significantly different and therefore the predictability assumption could not be confirmed. Furthermore, they found a very weak determination coefficient on the regression model, which was set up to predict pig odour ratios based on n-butanol ratios. The results of our study are also in line with the results of McGinley and McGinley (2010) who did not find a direct correlation between the individual thresholds of panellists for n-butanol and for H_2S. Our results also agree with the results of Parker et al. (2005) who found a small correlation between individual n-butanol and individual feedlot odour detection thresholds. Parker et al. (2005) concluded from several years of experience with odour measurements that n-butanol sensitivity is often weakly correlated to feedlot odour sensitivity.

4.5 Conclusions

Of the 4 investigated sources of variation, repetitions made by individual panellists were the most important for n-butanol as well as for pig house odour. However, repetitions had no significant effect. This suggests that the overall variation on odour thresholds for both odour types was small.

It appeared that the intra-panellist repeatability was better for pig house odour than for n-butanol, since the median coefficient of variation for n-butanol repetitions was almost twice that for pig house odour repetitions. However, statistically no significant difference could be found, suggesting a similar intra-panellist repeatability for n-butanol and for pig house odour. No significant difference was found between the median coefficient of variation on the results of groups of qualified panellists for n-butanol and for pig house odour samples. From this experiment, the inter-panellist repeatability could therefore be considered as alike for n-butanol and for pig house odour.

From these results, one could conclude that the odour measurements performed by the panellists were repeatable both for n-butanol and for pig house odour.

A weak relation was found between the panellists' n-butanol sensitivity and their sensitivity to pig house odour. Therefore, the assumption of CEN (2003), namely that the sensitivity for the reference will be a predictor for the sensitivity to other substances (CEN, 2003), could not be confirmed in this study focussing on pig house odour.

Annexe

The pig odour ratio (Y_{env}) and n-butanol ratio (Y_{nbut}) in the study of Defoer & Van Langenhove (2004) were defined as:

$$Y_{env} = \frac{x_{p,i}^{env}}{x_p^{env}} \qquad \text{Eq. (A.1)}$$

$$Y_{nbut} = \frac{x_{p,i}^{nbut}}{x_p^{nbut}} \qquad \text{Eq. (A.2)}$$

where $x_{p,i}^{env}$ and $x_{p,i}^{nbut}$ were the individual detection thresholds for a pig house odour and an n-butanol sample, respectively, measured by the i^{th} panellist (i = 1,2,3,…,n) in the p^{th} panel

and where x_p^{env} and x_p^{nbut}were the odour concentration (panel detection threshold) of a pig house odour and an n-butanol sample, respectively, measured by the p^{th} panel consisting of n panellists.

CHAPTER 5

INVESTIGATING MEASURES TO IMPROVE THE PRECISION OF AN ODOUR LABORATORY PERFORMING DYNAMIC OLFACTOMETRY

Redrafted from:

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CHAPTER 5: INVESTIGATING MEASURES TO IMPROVE THE PRECISION OF AN ODOUR LABORATORY PERFORMING DYNAMIC OLFACTOMETRY

The results of interlaboratory comparison tests (Mannebeck and Maxeiner, 2009) show that insufficient laboratories fulfil the requirements of CEN (2003) regarding the accuracy and precision (repeatability) limit. The precision, unlike the accuracy (chapter 1), can be assessed for both environmental odours, such as pig house odour as for the reference gas, n-butanol. In this chapter, the possibility of improving the precision of an olfactometric laboratory for both pig house odour and n-butanol measurements will be investigated.

5.1 Introduction and scope

Dynamic olfactometry is widely applied within Europe to measure odour concentrations and emissions from agricultural sources. The large implications of olfactometric results on stakeholders, which are in the first place farmers and neighbouring residents, emphasise the importance of reliable measurements. To obtain a reliable sensor, CEN (2003) sets two selection criteria for the assessors, namely an individual repeatability criterion and an individual sensitivity criterion (CEN, 2003) (see chapter 3). These panel selection criteria are evaluated during measurements with the reference gas, n-butanol. CEN (2003) then assumes that the quality of the panellists' performance for the reference gas is predictive for and transferable to their performance for environmental odours (see chapter 4). Next, to the requirements for the qualification of panellists, CEN (2003) also prescribes two quality requirements for laboratory performance: an accuracy criterion and a precision limit (see chapter 1.8.5). The definitions of both criteria are given in section 1.8.5. In summary:

- The accuracy (A_{od}) should be equal or lower than 0.217 and can only be assessed for the reference odour.
- The repeatability limit (r) should be below or equal to 0.477. The corresponding repeatability factor (10^r) therefore must be below or equal to 3 (CEN, 2003).

As shown in section 1.8.5, this limit value for the repeatability factor means that the difference between two consecutive single measurements on the same testing material in one odour laboratory under repeatability conditions should not be larger than a factor 3 in 95 % of the cases (CEN, 2003). To those who are not familiar with the variability associated with dynamic olfactometry (Laor et al., 2014) this precision limit might seem very large. In practice however, this repeatability limit is often not achieved (Laor et al., 2014; Van Harreveld et al., 2009; Munoz et al., 2010; Ubeda et al., 2013).

Next to variable results within a laboratory (McGinley & McGinley, 2006; Laor et al., 2014), large differences can also exist between the results of different laboratories (Table 1.13) when analysing replicate samples (Laor et al., 2014; Bereznicki et al., 2012; Hayes et al., 2014; Klarenbeek et al., 2014). These large differences are reflected in a poor reproducibility. This can pose problems, for example, when evaluating odour reduction techniques (Jonassen et al., 2012; Jonassen et al., 2014). The large variation in olfactometric results, within and between laboratories, does complicate a correct interpretation of results by stakeholders, such as farmers, neighbouring residents, policy makers, environmental regulatory agencies, researchers, manufacturers of abatement techniques, environmental consultants (Ubeda et al., 2013; Higuchi, 2009; McGinley, 2002; Munoz et al., 2010), while this is just of major importance.

To achieve results of high quality and to attain uniformity between laboratories, it is vital that individual laboratories invest in possible measures to improve the precision of their measurements. Individual laboratories can tackle different variables to enhance their precision e.g. the panel selection and training procedure (Mair et al., 2006; Capelli et al., 2010; Boeker et al., 2008; Zimmerman, 2015), the panel size (Mair et al., 2006; Boeker et al., 2008; Van Harreveld & Heeres, 1995; CEN, 2003; Brattoli et al., 2014; Ogink et al., 1995) and the number of repetitions per panellist (determined by the number of rounds per sample) (Van Harreveld & Heeres, 1995; Klarenbeek et al., 2014; Ogink et al., 1995; CEN, 2003).
Few scientific researches however, quantified the effect of different variables on the precision of an odour laboratory, in terms of repeatability (Van Harreveld & Heeres, 1995; Boeker et al., 2008; Boeker & Haas, 2007; Ogink et al., 1995).

Olfactometric measurements are labour-intensive and time-consuming. Therefore, simulating odour concentrations (Boeker et al., 2008; Ogink et al., 1995) can be a valuable alternative for investigating the effect of different variables on the precision of an odour laboratory.

In this study, an intensive simulation, using datasets of measured olfactometric thresholds, was performed to identify the measurement variables that significantly affect the laboratory's precision in terms of repeatability and to quantify their effects. The specific purposes were:

- To investigate the effects of panel size and of the number of panellist's thresholds on the precision of n-butanol and pig odour measurements
- To analyse the effect of the individual repeatability of panellists on the precision of n-butanol measurements
- To study the effect of a different odour type on the laboratory's precision

5.2 Materials and methods

The influence of different measurement characteristics on the precision of an odour laboratory was studied: 1) individual repeatability of panellists, 2) odour type, 3) panel size and 4) number of panellist's thresholds, determined by the number of rounds (ro.). Matlab was used to randomly constitute odour panels and to simulate odour concentrations based on ten datasets of olfactory thresholds, measured by CEN-qualified panellists for two types of odour, namely n-butanol and pig odour.

5.2.1 Used datasets

Two datasets were used to study the influence of the individual repeatability of panellists on the laboratory's precision for n-butanol (Table 5.1; analysis described in section 5.3.1). These datasets each contained 100 individual n-butanol thresholds, which were measured during a panellists' performance follow-up and using a certified calibration gas of 61.1 ppmv n-butanol in nitrogen (Westfalen AG, Münster, Germany) and a calibrated, 4-ports olfactometer with 'Yes-No' presentation mode (Odournet instrument TO8, Kiel, Germany). The datasets were named respectively the dataset of the "best" panellists and the dataset of the "good" panellists, depending on the panellists' performance level (PPL). The datasets of the best and of the good panellists, each consisted of 10 panellists and of 10 consecutively measured thresholds per panellist (Table 5.1). These consecutively measured thresholds per panellist were determined over different measuring sessions on different measuring days. Blanks were presented at altering positions within the different series of dilutions to introduce variation in the sequences presented to the panellists and also the start step could alter in between the series

presented to a panel. The panel selection criteria (1 and 2) of these panellists were calculated upon their 10 consecutive thresholds. x (panel selection criterion 1 = the individual repeatability) was calculated for each panellist according to CEN (2003), as the antilog of the standard deviation calculated from the logarithms (log10) of his/her 10 consecutively measured thresholds expressed in ppb n-butanol. y (panel selection criterion 2 = individual sensitivity was calculated for each panellist according to CEN (2003), as the mean from the logarithms (log10) of his/her 10 consecutively measured thresholds expressed in ppb n-butanol. The individual repeatability should be lower than 2.3 for panellist qualification and the individual sensitivity within the limits 20 – 80 ppb (CEN, 2003). The characteristics of both groups of panellists can be found in table 5.2. Both groups fulfilled both panel selection criteria of CEN (2003) (Table 5.2). They only displayed a different level of individual repeatability: the "best" panellists had on average an individual repeatability of 1.6 and the "good" panellists showed on average an individual repeatability of 2.0 (panel selection criterion 1) (Table 5.2). The individual sensitivities of the "best" and of the "good" panellists, following panel selection criterion 2 of CEN (2003), were comparable: the best panellists showed on average an odour threshold at an analytical concentration of 35 ppb n-butanol in nitrogen and the good panellists had on average an odour threshold at an analytical concentration of 38 ppb n-butanol in nitrogen (Table 5.2).

Table 5.1 Structure of the datasets for the comparison of both PPL

Odour	Dataset	PPL	N panellists [a]	N od. Thresholds [b]
n-butanol	1	Best	10	100
	2	Good	10	100

[a] Total number (N) of panellists
[b] Total number (N) of individual odour thresholds

Another eight datasets were used to study the influence of odour type on the laboratory's precision (Table 5.3; section 5.3.2). The experimental data for these 8 datasets were gathered over 4 measuring days, on which 12 panellists measured both one n-butanol and one pig odour sample in 6 rounds per measuring day. In total, 18 different panellists participated in the measurements over these 4 days. These measurements were performed during the measurement campaign described in Hove et al. (2016), using a calibrated, 4-ports olfactometer with 'Yes-No' presentation mode (Odournet instrument TO8, Kiel, Germany) and 19 CEN-qualified panellists.

Table 5.2 Ranges of the panellists' results for the selection criteria of CEN (2003), measured during the panellists' performance follow-up.

Panel selection criteria	PPL	
	Good	***Best***
CEN Crit. 1 (x ≤ 2.3) [-]		
minimum	1.92	1.43
maximum	2.20	1.83
average	2.03	1.63
CEN Crit. 2 (20 ≤ y ≤ 80]) [ppb]		
minimum	21	23
maximum	56	69
average	38	35

Table 5.3 Structure of the datasets for the comparison of both odour types

Odour	Dataset	N days [a]	N panellists / day [b]	N od. Thresholds [c]
n-butanol	3, 4, 5, 6	4	12	288
pig odour	7, 8, 9, 10	4	12	288

[a] Total number (N) of measuring days
[b] Total number (N) of panellists measuring per day
[c] Total number (N) of individual thresholds (over the 4 datasets)

Each of the 8 datasets consisted of 72 thresholds for one odour sample, whether n-butanol or pig odour, that had been measured by 12 panellists in 6 rounds on one measuring day. Blanks were presented at altering positions within the different rounds to introduce variation in the sequences presented to the panellists. The starting step could also alter for the different rounds. The panellists which measured these samples, were found qualified according to CEN (2003), based on their last 10 individual threshold estimates for n-butanol, determined before the measurement campaign started (Hove et al., 2016).

All 10 above mentioned datasets, were used also to study the influence of the panel size and of the number of individual thresholds per panellist (determined by the simulated number of dilutions' rounds presented during a measurement), see section 5.2.2.

A statistical analysis of the variance components of these primary datasets (table 5.1, table 5.3) can be found in appendix B (table B.1, table B.2).

5.2.2 Development of the precision analysis tool

A precision analysis tool was developed in Matlab (Matlab 8.6, The MathWorks Inc., Massachusetts, USA). This tool was used to randomly assemble a predefined number (n) of odour panels of a requested panel size using the datasets described in section 2.1. Panels consisting of 4, 5, 6, 7 and 8 panellists were assembled. A minimum panel size of 4 was chosen according to CEN (2003). For the comparison of panels composed of best panellists and of panels composed of good panellists, the n of randomly constituted panels, requested per panel size, was set at 45. The number of simulated panels per panel size for the comparison of the precision for n-butanol and pig odour was set at 495. Both these numbers correspond respectively with the limiting number of possible combinations of panellists in the requested panel sizes (4 to 8). These limiting numbers were found at the highest panel size (8 panellists). Equal numbers of panel drawings per panel size allowed a direct comparison of the precision for all panel sizes.

It was implied in the tool that, when randomly assembling a panel, the same panellist could not occur twice in that panel. The Matlab-tool randomly selected 3 thresholds per panellist for each sample. The first two selected thresholds per panellist, served to do the analysis at the level of 2 rounds. All three selected thresholds per panellist, served to do the analysis representative for 3 rounds.

Per randomly constituted panel, the odour concentration was calculated in [$ou_E\ m^{-3}$] according to CEN (2003) and for 2 rounds and 3 rounds separately. Retrospective screening (r.s.) of the panellists' results was applied by the code on all samples and this according to CEN (2003). The odour concentration of a sample before r.s. was calculated as the geometric mean of all thresholds of all respective panellists. The ratios (ΔZ-values) between the panellists' individual thresholds (Z_{ITE}) and the panel's threshold ($\bar{Z}_{ITE}$, geometric mean) were calculated, following the r.s. procedure of CEN (2003), using the following equation:

$$\text{If } Z_{ITE} \geq \bar{Z}_{ITE} \text{ then } \Delta Z = \frac{Z_{ITE}}{\bar{Z}_{ITE}}$$

$$\text{If } Z_{ITE} \leq \bar{Z}_{ITE} \text{ then } \Delta Z = -\frac{\bar{Z}_{ITE}}{Z_{ITE}} \qquad \text{(Eq 5.1)}$$

The parameter ΔZ had to comply with $-5 \leq \Delta Z \leq 5$ (CEN, 2003). (Eq 5.2)

When one or more individual thresholds of one or more panellists deviated more than a factor 5 from the panel's threshold (could indicate less or more sensitive), all thresholds of the panellist with the largest ΔZ had to be eliminated from the calculation of the result

of the respective sample, according to CEN (2003). The geometric mean in that case had to be recalculated on the remaining thresholds and also the ΔZ values of the remaining panellists, again checking accordance with the ΔZ-rule (-5 ≤ ΔZ ≤ 5) of CEN (2003). The r.s. steps were repeated per sample till no deviations from the ΔZ-rule were found. The geometric mean found at that point, defined the result of that sample after r.s. and was given in the Matlab-output. An illustration of the retrospective screening procedure can be found in appendix A. The Matlab-output also gave the panel size after r.s.. This allowed to see whether the result of a sample after r.s. was valid regarding CEN (2003), because a minimum panel size of 4 panellists should remain. The number of deviating thresholds at the concentration before r.s. was also displayed in the Matlab-output.

The programming in Matlab resulted in a validated code for random panellists- and thresholds- compositions and for the calculation of odour concentrations according to CEN (2003). The great advantage of the code is that a large number of panel thresholds (odour concentrations) can be generated, based on experimental data. In this manner, multiple repetitions of samples can be produced, enabling to study the effect of different variables on the olfactometric precision.

5.2.3 Parameters for precision analysis

To study the effect of the different measurement variables on the precision of odour measurements, two parameters were calculated. Firstly, the coefficient of variation (CVlog) was calculated on the decimal logarithms of the odour concentrations simulated per dataset for a specific panel size (4,5, 6, 7 or 8) and a specific number of rounds (2 or 3). Secondly, the repeatability limit (r) (CEN, 2003) was calculated on the decimal logarithms of the odour concentrations simulated per dataset for a specific panel size (4,5, 6, 7 or 8) and a specific number of rounds (2 or 3). The repeatability limit was calculated according to CEN (2003), namely by the formula $r = t.\sqrt{2}.sr$, in which t is the value from the student t distribution corresponding with n-1 degrees of freedom and a 95 % confidence interval, and in which s_r is the repeatability standard deviation on the panel thresholds of n panels. The repeatability factor (10^r) was also calculated, representing the maximum factor between two consecutive repetitions of the same sample in 95 % of the cases. Invalid odour concentrations according to CEN (2003), obtained by less than 4 panellists after r.s., were not considered in the calculation of both precision parameters. The results for both CVlog and r as a function of panel size and the number of rounds were graphically represented for the different PPL and odour types. The calculated r

were also compared with CEN's repeatability limit, namely $r \leq 0.477$, corresponding with $10^r \leq 3$.

5.2.4 Statistical data-analyses

Regression models were set up for the precision parameters (CVlog and r) in SAS 9.3 (Proc. Mixed, SAS Institute Inc., NC, USA) as a function of the different measurement variables (PPL resp. odour type, panel size, number of rounds). In a first analysis (Table 5.4), the effect of the PPL, the panel size and the number of rounds on the repeatability of an odour laboratory was studied considering n-butanol only, using the results of the simulations on datasets 1 and 2 (Table 5.1). The regression model was set up as follows:

$$Ycv_{logi} = \beta_0 + \beta_n X_n + e_i \quad \text{(Eq 5.3)}$$

Ycv_{logi} and Yr_{logi} = precision parameters (dependent variables)

β_0 = intercept

β_n = regression coefficient for X_n

X_n = fixed effects, tested in the model (Panel size, PPL, Number of rounds)

I = measurement

e_i = residual

All variables were seen as categorical variables, namely the variable panel size existed of classes 4, 5, 6, 7 and 8; the PPL encompassed the classes 'good' and 'best' and finally the number of panellist's thresholds were represented by the classes 2 and 3 (Table 5.4). As an example, class 4 (Table 5.4), therefore contained the results of all simulated panels of panel size 4, so as well of the best as of the good panellists. Class 4 thus counted 90 panels (45 simulated panels per PPL times 2 PPL). The number of concentrations associated to a class also considered all the simulated concentrations for this class.

In a second analysis (Table 5.5), the effect of odour type, panel size and the number of rounds on the repeatability of an odour laboratory was studied, using the results of the simulations on 8 comparative datasets of n-butanol and pig odour. A mixed regression model of the precision parameter (dependent variable) was set up for that purpose, in which day was added as a random variable, since the 4 pig odour and the 4 n-butanol samples were sampled on 4 different days:

$$Ycv_{logij} = \beta_0 + \beta_n X_n + \mu_j + e_{ij} \quad \text{(Eq 5.4)}$$

Ycv_{logij} and Yr_{logij} = precision parameters (dependent variables)

β_0 = intercept

β_n = regression coefficient for X_n

X_n = fixed effects, tested in the model (Odour type, Panel size, Number of rounds)

μ_j = random fault on day level

I = measurement, j = day

eij = residual

In analogy with the first analysis, all variables were seen as categorical variables, namely the variable panel size existed of classes 4, 5, 6, 7 and 8; odour type consisted of the classes n-butanol and pig odour and finally the number of panellist's thresholds were represented by the classes 2 and 3 (Table 5.5). The number of concentrations associated to a class considered all the simulated concentrations for this class. For example class 5 (Table 5.5), contained all concentrations generated for panel size 5, using the 8 comparative (n-butanol and pig odour) datasets and by performing 2 and 3 rounds. The number of concentrations within class 5 in this second analysis therefore was 7920, i.e. 495 simulated panels per dataset times 8 datasets times 2 concentrations per panel (1 concentration for 2 rounds and 1 concentration for 3 rounds).

The effects of the different tested variables on the precision of the odour laboratory were studied by investigating their effect on both CVlog and r (see Eq 5.3 and 5.4) and this at a significance level of 0.05 ($p < 0.05$) (Tables 5.4 and 5.5). Significant differences ($p < 0.05$) in precision between the different panel sizes were checked by a Tukey post-hoc test. Interactions between the studied variables were also tested. Additionally, a linear model was set up for the precision of pig odour as a function of the panel size and of the number of rounds.

5.3 Results

93 % of the 40500 simulated odour concentrations were valid according to CEN (2003). So only 7 % of the simulated odour concentrations could not be used in the further analyses (Section 2.3). Results were considered as invalid, when the thresholds of less than 4 panellists remained after performing the retrospective screening procedure (according to CEN (2003)) on the respective thresholds. Invalid results were mainly found when simulating n-butanol measurements for panel size 4 during the second analysis.

5.3.1 Effects of the panellist's performance level, the panel size and the number of rounds on the laboratory's precision for n-butanol

Figure 5.1 presents both CVlog and r as a function of the panel size (4 to 8), the PPL ("good" or "best") and the number of panellist's thresholds (determined by the number of rounds performed during the measurement: 2 rounds or 3 rounds. From Figure 5.1, it can be seen that generally CVlog and r decrease with an increasing panel size. This is valid for both PPL (Fig. 5.1). CVlog and r are generally lower for 3 rounds compared to 2 rounds. (Fig. 5.1), suggesting a higher precision for 3 rounds. When comparing the results of best and of good panellists for the same panel size and the same number of rounds, it appears that the best panellists have a better precision than the good panellists (Fig. 5.1). Both PPL displayed results below the repeatability limit set by CEN (2003) (Fig. 5.1b) and therefore showed a higher precision than required by CEN (2003).

Table 5.4 shows the results of the final multilevel linear model representing the effects of the different tested categorical variables on the laboratory's precision for n-butanol. It can be seen that r was below the repeatability limit set by CEN ($r \leq 0.477$) for all variable classes and therefore CEN-fulfilling. The panel size ($p < 0.001$); the PPL ($p < 0.001$) as well as the number of rounds ($p < 0.001$) had significant effects on CVlog and on r (Table 5.4).

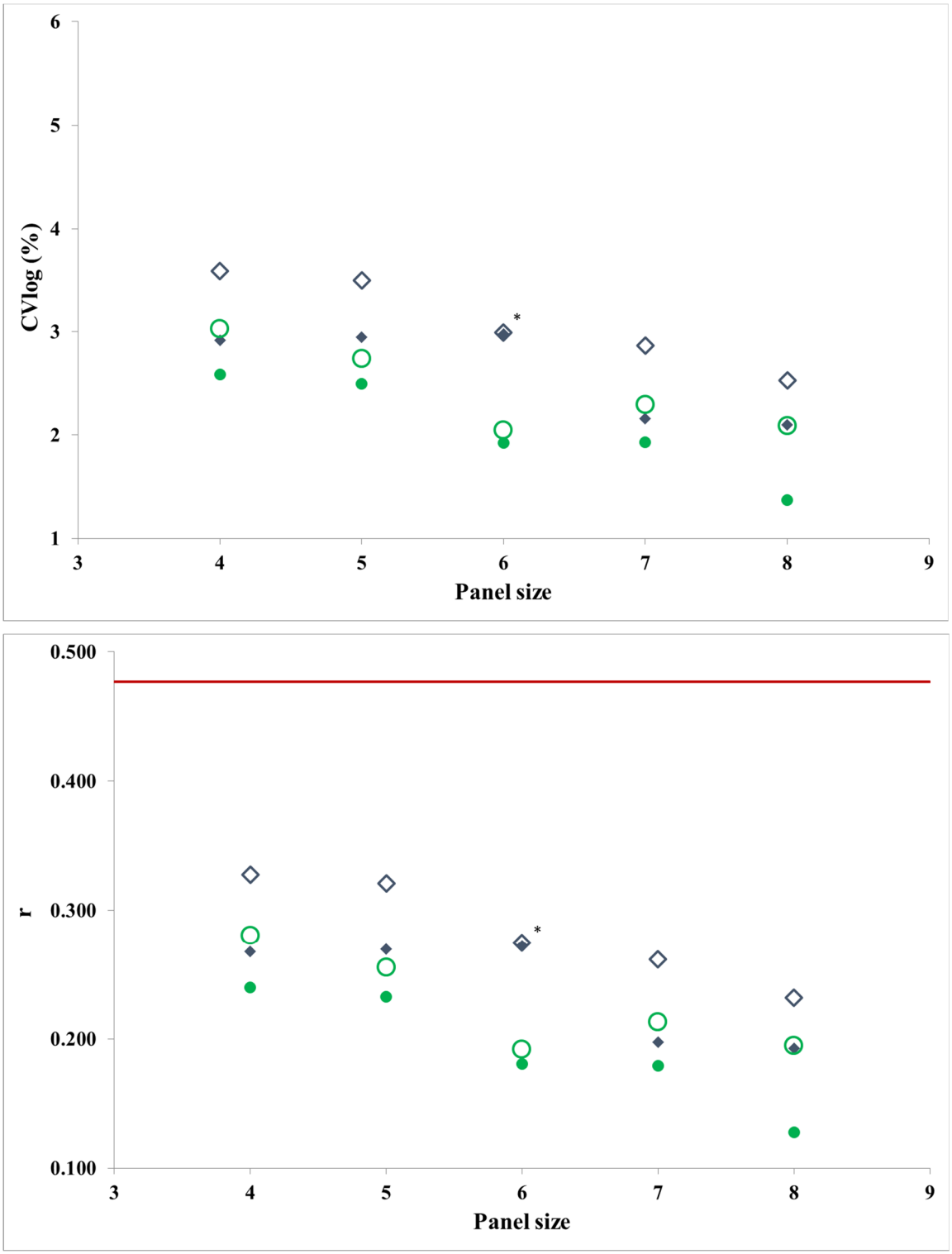


Figure 5.1 (a) Evolution of CVlog and (b) r as a function of the panel size, the PPL and the number of rounds, representing good panellists performing 2 rounds (◇), good panellists performing 3 rounds (◆), best panellists performing 2 rounds (○) and best panellists performing 3 rounds (●). The repeatability limit set by CEN is also displayed (—)(b). Slight differences (*) in CVlog and in r were found between 2 and 3 rounds for panels consisting of 6 good panellists, namely a CVlog of 2.991 (2 rounds) versus 2.974 (3 rounds) (Fig. 1a) and an r of 0.275 (2 rounds) versus 0.272 (3 rounds) (Fig. 1b).

In general, considering the pooled results of both PPL, the precision of panels improved with an increasing panel size, as both CVlog and r diminished with an increasing panel size (Table 5.4). Significant differences were found between the panel sizes 4 and 6 ($p = 0.027$ and 0.028 resp.), between 4 and 7 ($p = 0.004$ and 0.003 resp.), between 4 and 8 ($p < 0.001$), between 5 and 7 ($p = 0.013$ and 0.010 resp.) and between 5 and 8 ($p < 0.001$), analysing the effects on CVlog and on r respectively. In table 5.4, these significant differences are indicated by different letters for the respective panel sizes. Between other panel sizes no significant differences were found statistically. Based on these significant differences in precision between the concerning panel sizes and the corresponding changes (%) in the estimated r (Table 5.4), it follows that going from a panel size of 4 to 5 does not improve the precision significantly. Moving from a panel size of 4 towards 6 however significantly improves the precision (Table 5.4). From a panel size of 7 on, there is a significant improvement in precision compared to the panel sizes 4 and 5 (Table 5.4). There is no significant difference between the panel sizes 7 and 8 (Table 5.4), but a panel size of 8 realises the largest improvement in precision (33 %) (Table 5.4). The precision thus can be improved by moving from a minimum panel size of 4 towards a panel size of 6, 7 or 8. Enlarging the panel size from 4 to 8, changed the estimated r from 0.279 to 0.187 (Table 5.4), corresponding with a change in 10^r from 1.90 to 1.54. Those values of r and 10^r fulfil the laboratory performance requirements of CEN (2003), namely an r below or equal to 0.477 and 10^r below or equal to 3.

Panels composed of panellists with a higher individual repeatability, namely the “best” panellists showed a significantly higher precision than panels composed of good panellists, representing an improvement in the estimated r from 0.262 to 0.210 (Table 5.4), corresponding with an improvement of 10^r from 1.83 to 1.62. Performing 3 rounds instead of 2 rounds induced a significantly higher precision, giving an estimated r of 0.216 instead of 0.255 (Table 5.4) corresponding with a 10^r of 1.64 instead of 1.80. Doubling the panel size improved the precision for n-butanol the most (Table 5.4), when comparing the effects of the different tested variables (PPL, panel size, number of rounds) on the estimated CVlog and r. No significant interactions were found between the panel size and the PPL. Also no significant interactions appeared between the panel size and the number of rounds, nor between the PPL and the number of rounds.

Table 5.4 Final multilevel linear model describing the effects of different categorical variables on the precision for n-butanol.

Independent variable	No. of panels[1]	No. of conc.[2]	Precision parametres											
			CVlog (%)						*Repeatability limit r (CEN: r ≤ 0,477)*					
			β[3]	St. Error[4]	p value[5]	LSM[6]	Diff.[7]	Change (%)[8]	β	St. Error	p value	LSM	Diff.	Change (%)
Intercept (Constant)			3.55	0.12					0.325	0.010				
Panel size					< 0,001						< 0,001			
4	90	180	Ref. [9]	—		3.03	a	—	Ref.	—		0.279	a	—
5	90	180	-0.11	0.14		2.92	ab	-4	-0.009	0.012		0.270	ab	-3
6	90	180	-0.55	0.14		2.48	bc	-18	-0.049	0.012		0.230	bc	-18
7	90	180	-0.72	0.14		2.31	c	-24	-0.066	0.012		0.213	c	-24
8	90	180	-1.01	0.14		2.02	c	-33	-0.092	0.012		0.187	c	-33
PPL					< 0,001						< 0,001			
Good	225	450	Ref 2 [10]	—		2.86	d	—	Ref 2	—		0.262	d	—
Best	225	450	-0.61	0.09		2.25	e	-21	-0.052	0.008		0.210	e	-20
Number of rounds					< 0,001						< 0,001			
2	450	450	Ref 3 [11]	—		2.77	f	—	Ref 3	—		0.255	f	—
3	450	450	-0.43	0.09		2.34	g	-15	-0.039	0.008		0.216	g	-15

[1] Total number of panels associated with variable
[2] Total number of concentrations associated with variable
[3] Regression coefficient associated with variable
[4] Standard error on regression coefficient
[5] p-value associated with variable
[6] LSM = Estimate of the precision parameter (CVlog resp. r) from least square means
[7] Differing letters indicating significant differences between the respective variable classes
[8] Change (%) in the LSM compared to the reference
[9] Reference for the panel size
[10] Reference for the panellists' performance level
[11] Reference for the number of rounds

5.3.2 Effects of odour type, panel size and the number of rounds on the laboratory's precision

Figure 5.2 represents the average CVlog and r, calculated over the 4 measuring days, as a function of the panel size (4 to 8), the odour type (n-butanol or pig odour) and the number of panellist's thresholds (determined by the number of dilutions' rounds performed during the measurement (2 or 3). For pig odour, the average CVlog and r linearly decrease with increasing panel size, indicating an improvement in precision with an increasing panel size (Fig. 5.2). For n-butanol, the average CVlog and r slightly increase till panel size 6. From a panel size 6 on, CVlog and r linearly go down for n-butanol (Fig. 5.2). Furthermore, CVlog and r are lower for pig odour than for n-butanol, considering all panel sizes and all number of rounds (Fig. 5.2). A higher precision thus is achieved for pig odour than for n-butanol. CVlog and r are lower for 3 rounds compared to 2 rounds and this for both odour types (Fig. 5.2), suggesting a better precision for 3 rounds. In addition, the r of both odour types are always below the limit value defined by CEN (2003), namely $r \leq 0.477$ (Fig. 5.2).

Table 5.5 shows the results of the final multilevel linear model presenting the effects of the different tested categorical variables on the laboratory's precision. The estimated r were always below the limit value set by CEN (Table 5.5). It was found that the panel size ($p < 0.001$) as well as the odour type ($p < 0.001$) and the number of rounds ($p = 0.020$ resp. $p = 0.009$) had significant effects on CVlog and on r (Table 5.5). Panels showed a significantly higher precision for pig odour than for n-butanol with an estimated r of 0.238 for pig odour compared to an r of 0.410 for n-butanol (Table 5.5), corresponding with a 10^r of 1.73 for pig odour and a 10^r of 2.57 for n-butanol. These values for pig odour and for n-butanol can be compared with the limits set by CEN (2003), namely $r \leq 0.477$ and $10^r \leq 3$. Considering the pooled pig odour and n-butanol results, the precision of panels enhanced with an increasing panel size, as both CVlog and r decreased with an increasing panel size (Table 5.5). Significant differences were found between the panel sizes 4 and 8 ($p < 0.001$), between 5 and 8 ($p < 0.001$) and between 6 and 8 ($p = 0.002$ resp. $p < 0.001$), analysing the effects on CVlog and on r respectively (Table 5.5). Between the panel sizes 4 and 7 ($p = 0.087$ resp. $p = 0.028$) and between the panel sizes 5 and 7 ($p = 0.069$ resp. $p = 0.028$) significant differences ($p \leq 0.05$) were found based on r and nearly significant differences ($p \leq 0.10$) based on CVlog respectively. Based on the given significant differences between panel sizes and the respective changes (%) in the estimated repeatability limit (Table 5.5), it follows that going from a panel size of 4 to a panel size of 5 or 6 does not improve the precision of the odour laboratory significantly (Table 5.5). From a panel size of 7 on, there is a significant improvement in precision

compared to panel sizes 4 and 5 based on the evolution in r (Table 5.5). A panel size of 8 realises a significant improvement in precision, compared to panel sizes 4, 5 and 6 (Table 5.5).

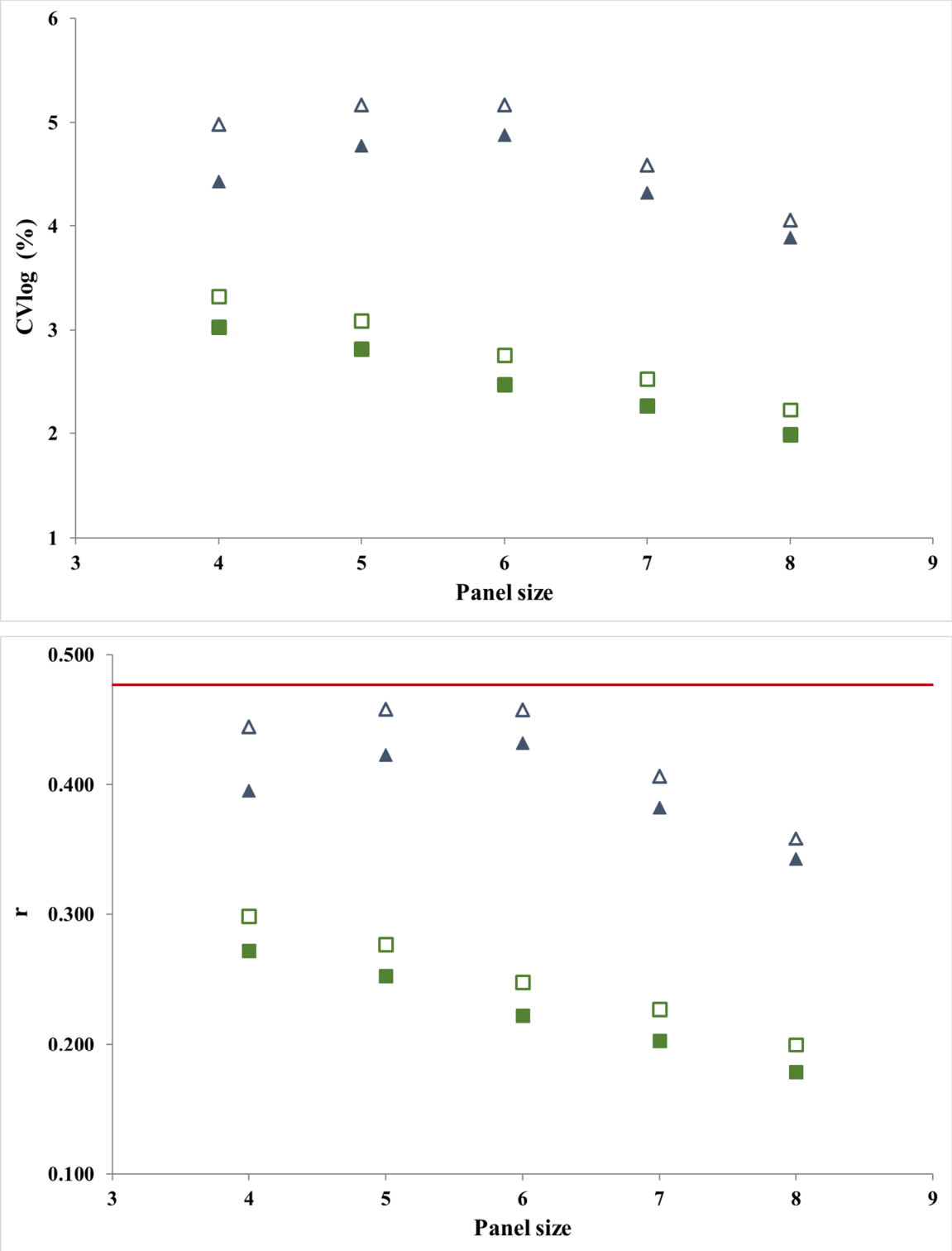


Figure 5.2 (a) Evolution of CVlog and (b) r as a function of the panel size, the odour type and the number of ro., representing n-butanol and 2 rounds (△), n-butanol and 3 rounds (▲), pig odour and 2 rounds (□), pig odour and 3 rounds (■). The repeatability limit of CEN (—) is also displayed (b).

Table 5.5 Final multilevel linear model describing the effects of different categorical variables on the precision for n-butanol and pig odour.

Independent variable	No. of panels[1]	No. of conc.[2]	Precision parametres											
			CVlog (%)						*Repeatability limit r (CEN: r ≤ 0,477)*					
			β[3]	St. Error[4]	p value[5]	LSM[6]	Diff.[7]	Change (%)[8]	β	St. Error	p value	LSM	Diff.	Change (%)
Intercept (Constant)			5.08	0.21					0.452	0.015				
Panel size					< 0,001						< 0,001			
4	3960	7920	Ref.[9]	—		3.94	a	—	Ref.	—		0.353	a	—
5	3960	7920	0.02	0.20		3.96	a	1	0.000	0.016		0.353	a	0
6	3960	7920	-0.12	0.20		3.82	a	-3	-0.013	0.016		0.340	ab	-4
7	3960	7920	-0.51	0.20		3.43	ab	-13	-0.048	0.016		0.305	bc	-14
8	3960	7920	-0.90	0.20		3.04	b	-23	-0.083	0.016		0.270	c	-23
Odour type					< 0,001						< 0,001			
n-Butanol	9900	19800	Ref 2[10]	—		4.63	c	—	Ref 2	—		0.410	d	—
Pig Odour	9900	19800	-1.98	0.13		2.65	d	-43	-0.172	0.010		0.238	e	-42
Number of rounds					0.020						0.009			
2	19800	19800	Ref 3[11]	—		3.79	e	—	Ref 3	—		0.338	f	—
3	19800	19800	-0.30	0.13		3.49	f	-8	-0.027	0.010		0.310	g	-8

[1] Total number of panels associated with variable
[2] Total number of concentrations associated with variable
[3] Regression coefficient associated with variable
[4] Standard error on regression coefficient
[5] p-value associated with variable
[6] LSM = Estimate of the precision parameter (CVlog resp. r) from least square means
[7] Differing letters indicating significant differences between the respective variable classes
[8] Change (%) in the LSM compared to the reference
[9] Reference for the panel size
[10] Reference for the odour type
[11] Reference for the number of rounds

The precision thus can be improved by moving towards a panel size of 7 or 8. Increasing the panel size from 4 to 8, moved the estimated r from 0.353 to 0.270 (Table 5.5), corresponding with a change in 10^r from 2.25 to 1.86. These values fulfil the CEN-requirements, namely $r \leq 0.477$ and $10^r \leq 3$. Table 5.5 shows that enhancing the number of rounds from 2 to 3 had a significant effect on the precision of panels, displaying an estimated r of 0.310 instead of 0.338, corresponding with a 10^r of 2.04 instead of 2.18. No significant interactions were found between panel size and odour type. Also no significant interactions appeared between odour type and the number of rounds, nor between the panel size and the number of rounds.

Specifically, for pig odour, a clear linear relation could be seen between the precision (CVlog and r) and an increasing panel size for both 2 rounds and 3 rounds (Fig. 5.2). Examining the effects of these variables for pig odour specifically, showed that CVlog diminished with 0.269 for one extra panellist and and with the same amount for one extra round (performing 3 rounds instead of 2 rounds). The same effect was seen on r for pig odour, namely r decreased with 0.024 for one extra panellist and with the same amount for one extra round (performing 3 rounds instead of 2 rounds).

5.4 Discussion

Invalid results (7 % of the simulated odour concentrations) due to a panel size lower than 4 after r.s. mainly occurred when starting at a panel size of 4 and exceptionally at a panel size of 5. This is in accordance with CEN (2003) which assumes that generally no more than one third of a panel will show deviant sensory behaviour during a measurement. The possibility of drop out of panellists, due to temporary deviating responses detected by retrospective screening, should however be considered when determining the starting panel size for an analysis. Specifically, starting from a panel size of 4 panellists holds a risk for invalid results (after r.s.). As mentioned by Brattoli et al. (2014), starting from panel sizes higher than 4 is also beneficial since the number of panellists with valid responses both influence the precision of olfactometric measurements and the quality of the results.

For n-butanol, the average CVlog and r slightly increased till panel size 6, while for pig house odour the average CVlog and r already decreased at panel size 5 (Figure 5.2). The increase in CVlog and r, noticed for n-butanol, could be a side-effect of applying the retrospective screening procedure according to CEN (2003) resulting in lower panel sizes (4 or 5) at the end of a measurement compared to the panel size at the start of the measurement (5 or 6). Since the variation appeared to be larger when measuring n-

butanol than when measuring pig odour, the effect of the retrospective screening procedure could possibly have more influence the evolution for n-butanol.

The PPL, the panel size and the number of rounds all significantly affected the precision of odour panels for n-butanol (Table 5.4). From them, doubling the panel size of 4 to 8 had the largest effect on the precision of odour panels, but panel sizes 6 and 7 also induced a significant improvement in precision (Table 5.4). The effect of doubling the panel size was larger than the effect of a more strengthened panel selection procedure (smaller crit. 1) (Table 4). These results are in accordance with Ogink et al. (1995), who found that doubling the panel size from 8 to 16 clearly improved the precision and that a more severe panel selection was not so effective. Boeker et al. (2008) also found that larger panel sizes diminished the measurement uncertainty considerably next to strengthening the panel selection based on criterion 1. The improvement induced by an extra round was nearly the same as by strengthening the panel selection procedure (Table 5.4). Klarenbeek et al. (2014) also mentioned that increasing the panel size is more effective for improving the precision than a higher number of measurements. No significant interactions appeared between the panel size and the PPL. Also no significant interactions were found between the panel size and the number of rounds, nor between the PPL and the number of rounds.

The odour type, the panel size and the number of rounds all significantly affected the laboratory's precision for n-butanol and pig odour (Table 5.5). From these three variables, the odour type had the largest influence. A significantly better precision was achieved for pig odour compared to n-butanol and this was deduced from 39600 simulated odour concentrations (Table 5.5). The inter-panellist and intra-panellist repeatability already seemed better for pig odour than for n-butanol in the study by Hove et al. 2016. In this study a significant difference between the inter-panel repeatability (precision) for pig odour and that for n-butanol could actually be found (Table 5.5). The results of this simulation are in line with the study of Sneath (2003) who also found a better repeatability factor for real odours (pig odour and restaurant odour) than for n-butanol. Van Harreveld and Heeres (1995) also found a lower spread on results for environmental odours compared to n-butanol, but the difference was non-significant. Laska and Hudson (1991) also detected lower variabilities for multi-component mixtures compared to the unmixed odorants. They suggested that odour mixtures rather than single substances should be used as a reference gas in order to reduce variability. CEN (2003) also noted that a reference mixture would be preferable, because fundamental research and inter-laboratory comparisons of environmental odours and n-butanol had

shown that the repeatability limit for odour mixtures is better than that for single compounds. The results from these inter-laboratory studies are in agreement with the simulated results of this study.

The odour type under analysis, however, is a fixed condition of a measurement. After odour type, doubling the panel size had the largest effect on the precision of odour panels compared to more rounds, considering the pooled n-butanol and pig odour results (Table 5.5). A panel size of 7 also induced a significant improvement in precision and had a higher effect on the precision than more rounds, considering the pooled n-butanol and pig odour results (Table 5.5). No significant interactions were detected between panel size and odour type. Also no significant interactions were found between odour type and the number of rounds, nor between the panel size and the number of rounds.

Based on the presented models and considering the effects of panel size, the number of rounds and the PPL on the precision and since odour type is a fixed condition of a measurement, it seems that the largest improvement in precision can be generated by enlarging the panel size: specifically by moving towards a panel size of 7 or 8 panellists a significant improvement in precision can be realised (Table 5.4 and 5.5). This is valid considering n-butanol and pig odour (Table 5.4 and 5.5). This is in accordance with Klarenbeek et al. (2014) which stated that increasing the number of panellists per measuring session had more effect on the precision of odour measurements than a higher number of repetitions. CEN (2003) also recommended higher panel sizes than 4 after r.s. to improve the repeatability and accuracy of measurements. Using a higher number of panellists for improvement of the precision is also in accordance with the study of Clanton et al. (1999), who found a lower variation on results when using 8 panellists and performing 1 round, compared to using 4 panellists and performing 2 rounds.
Enhancing the panel size surely increases the costs of olfactometric measurements. Depending on the application of the olfactometric measurements and the desired precision, laboratories could choose for a combination of the investigated measures to improve their precision. Specifically for pig odour, linear analysis of the results showed that the effect of one extra round on the precision, namely when going from 2 to 3 rounds, was comparable with the effect of one extra panellist.

The repeatability limit for n-butanol measurements was lower in the first analysis (Fig. 1) than in the second analysis (Fig. 2) and fulfilled in both cases the repeatability limit prescribed by CEN (2003). The data used in the first analysis consisted mainly of panellists' performance follow-up measurements preceding the measurement campaign described in Hove et al. (2016) (Chapter 4), which on its turn provided data for the second

analysis. 18 of the 20 panellists represented the first analysis, participated in the measurements used in the second analysis. The difference in panellists' performance between both analyses is possibly due to the variability of the human sense of smell in time. The first data are more representative for panel selection measurements, while the second data are representative for practice measurements compiling longer measurement sessions and measurements performed at larger time-intervals.

The precision for pig odour was significantly higher than that of n-butanol (Fig 5.2 and Table 5.5). In the study of Hove et al. (2016) a low predictability of n-butanol sensitivity for pig odour sensitivity was detected. These differences in performance characteristics for n-butanol (a single compound) and pig odour (an odour mixture) could raise questions about the use of n-butanol for the selection of odour panels with the purpose of measuring pig odour samples. More specifically, does panel selection with n-butanol lead to a representative panel for performing pig odour measurements?

5.5 Conclusions

An extensive simulation on olfactometric data was performed to study the effect of different variables on the precision of an odour laboratory. Firstly, the effects of the panellists' performance level, the panel size and the number of rounds were studied on the precision of an odour laboratory for n-butanol. Secondly, the effects of odour type, panel size and the number of rounds were investigated on the laboratory's precision for both n-butanol and pig odour.

The first analysis showed that all three tested variables had a significant effect on the precision for n-butanol. The effect of an increasing panel size was the highest. In the second part of the study, also all three tested variables had a significant effect on the laboratory's precision. Odour type had the largest influence, followed by a larger panel size.

From both parts of the study could be deduced that investing on panel size is a good methodological means to improve the laboratory's precision. For pig odour specifically, the effect of an extra round (3 rounds instead of 2 rounds) was similar to the effect of an extra panellist, giving an extra means to enhance the precision. The precision for pig odour was significantly higher than that of n-butanol, which could raise questions about the use of n-butanol for panellists' selection to measure pig odour samples.

CHAPTER 6

GENERAL DISCUSSION

CHAPTER 6: GENERAL DISCUSSION

Odour emissions and odour nuisance generated by pig production have gained increased attention in the past years. The most-commonly applied method in Europe to measure odour concentrations emissions from agricultural sources is dynamic olfactometry, according to the standard EN 13725:2003. The applicability of the CEN (2003) standard for assessing livestock odour emissions however had not been evaluated yet.

In this PhD-thesis, the purpose was to evaluate the applicability of the CEN (2003)-standard for pig house odour measurements based on a critical literature review and by performing odour measurements and simulations (Chapters 3 to 5). The second aim was to investigate possible measures to optimise the method (Chapter 5).

This chapter starts with a discussion of the research findings on the precision of dynamic olfactometry and on the representativeness of the current reference gas, together with their implications for practice. The advantages of performing odour simulations are also presented. The general discussion ends with situating dynamic olfactometry amongst other odour measurement techniques used in odour assessments.

6.1 Measurement uncertainty and implications for practice

The large measurement uncertainty, related to dynamic olfactometry (Boeker et al., 2008; Zarra et al., 2008) can cause problems in the determination of odour emission factors (Mielcarek & Rzenik, 2015) and while evaluating the efficiency of odour abatement techniques (Jonassen et al., 2012; Hansen et al., 2014; Jonassen et al., 2014; Ubeda et al., 2013). Large differences between the results of different laboratories for replicate samples result from that: differences up to a factor 7 were found between the results of different laboratories for swine odour samples by Berezneki et al. (2012).

However, information on the different sources of variation within laboratories was limited. Only a few studies investigated these sources of variation, using different approaches (Van Harreveld & Heeres, 1997; Clanton et al., 1999; Klarenbeek et al., 2014). Therefore, in this Phd work, the influence of the following aspects of variation were investigated: differences between samples, between panels, between panellists and between

repetitions of the same panellist (Chapter 4). The individual variability of panellists in time appeared to be the largest source of variation as well for n-butanol as for pig odour (Chapter 4). This result was in line with the results of previous scientific experiments (Van Harreveld & Heeres, 1997; Clanton et al., 1999), where the intra-panellist variability was found to be the largest contributor to the measurement variability (next to the inter-panellist variability). In our study the inter-panellist variability appeared to be smaller than the intra-panellist variability (Chapter 4). The results of the variance study performed by Klarenbeek et al. (2014) could not be compared with our results, because the intra-panellist and inter-panellist variation were not included in that study.

Aiming to reduce the large measuring uncertainty of dynamic olfactometry and considering this pre-knowledge (Chapter 4), the influence of the following measures was investigated in depth in the view of improving the olfactometry precision (Chapter 5):

- Effect of a higher individual repeatability of panellists (panel selection criterion 1)
- A larger panel size
- A higher number of rounds

The effect of the odour type (pig odour compared to n-butanol) on the precision of odour measurements was also looked at (Chapter 5).

Both the panellists' performance level (concerning the individual repeatability), an increasing panel size, an increasing number of rounds and the odour type significantly affected the precision of odour measurements (Chapter 5).
Our results showed that:

- Working with a larger panel was more effective in increasing the precision than applying more rounds and also than a more stringent panel selection procedure (panel selection crit. 1).
- When considering both n-butanol and pig odour assessments, the odour type had the largest effect on the olfactometry precision and secondly a larger panel size. However, odour type is a fixed characteristic of an emission and therefore it is not an adaptable measurement variable.

In summary, to enhance the precision of an individual odour laboratory and in that way positively influence the reproducibility between laboratories, it is advisable to use a larger panel size than 4 (ideally 7 or 8) (Chapter 5), both for pig odour and for n-butanol measurements. A panel size of 4 already showed to be within the precision limit defined by CEN (2003) (CEN requires a repeatability factor lower than 3 and here the repeatability factor was 2.25 considering the pooled n-butanol and pig odour results)

(Chapter 5). However, working with panels of 4 panellists encloses a high risk for invalid results after retrospective screening: an occurrence of 11 to 50 % of invalid results was found when measuring with 4 panellists, considering the simulated n-butanol and pig odour results from Chapter 5. The repeatability factor improved from 2.25 (at panel size 4) towards 2.02 (at panel size 7) and resp. 1.86 (at panel size 8) with an increasing panel size (Chapter 5). The complementary advantage was that the occurrence of invalid results was much smaller at panel sizes 7 and 8, namely 1 % to 4 % of all simulations. The large and positive influence of an increasing panel size on the precision of odour measurements was also detected by Ogink et al. (1995); by Boeker et al. (2008) and by Klarenbeek et al. (2014). CEN (2003) also indicated that using a larger panel for measuring odours could lead to more reliable results, as well in terms of accuracy as in terms of precision.

The precision for pig odour was found to be significantly higher than that of n-butanol. The repeatability factor for pig odour was estimated to be 1.73 versus 2.57 for n-butanol, considering the pooled results of panel sizes 4 to 8 and both 2 and 3 rounds (Chapter 5). This difference between n-butanol and pig odour corresponded with the findings of Sneath (2003), who also found a better repeatability factor for real odours (pig odour and restaurant odour) than for n-butanol. For pig odour specifically, it was also seen that adding one extra panellist (in the range of 4 to 8 panellists) had the same effect on the olfactometry's precision as going from 2 to 3 dilutions' rounds (Chapter 5).

The application of the proposed precision enhancing measures however increases the costs and extends the duration of odour measurements. The personnel costs during a single measurement of one odour sample with 4 panellists in 2 rounds (minimum requirements of CEN (2003)) and using the Yes/no mode on a custom 4-ports olfactometer and considering a measuring duration of 10 minutes (cost of a panellist / hour: 28.35 Euros; cost of the operator / hour: 31.91 Euros) namely consist of 24.22 Euros. The costs displayed here do not include the sampling costs (material, transportation) nor the costs for the preparation of the sampling (by a sampling technician) nor the costs for the preparation of the measurement or interpretation of the measurement data (by the operator) nor the costs associated with the facility or calibration costs. As such, the calculated amounts can appear relatively low compared to the costs for olfactometric analyses performed by external laboratories (100 to 300 Euros) (personal communication). However, in this assessment and the following, the purpose is only to evaluate the increase in costs associated with a larger panel size

and/or a higher number of rounds. A further detailing of the other costs associated with dynamic olfactometry is given in Chapter 3.

Increasing the panel size and the number of rounds will extend the duration and increase the costs of the odour measurement of one sample as follows:

- When assessing the sample with 5 to 8 panellists compared to a panel size of 4, the measuring duration will double because only 4 panellists can measure simultaneously on the 4-ports olfactometer and thus a second session with the 5th to 8th panellist is required.
- For a single sample and using a regular 4-ports olfactometer, the additional cost of an extra panellist, e.g. when going from 4 to 5 panellists and considering 2 rounds, comes down to 41.5 % of the original personnel measuring costs. This is 10,04 Euros extra, namely 4.73 Euro extra for the panellist and 5.32 Euro extra for the operator (2nd measuring session is needed). When going from 5 to 6 or from 6 to 7 or from 7 to 8 panellists and considering 2 rounds, the additional personnel measuring cost per sample would only be 4.73 Euros since a 2nd measuring session is inherent from 5 panellists onwards and thus the operator will already be present (included in the costs).
- When performing 3 rounds instead of 2 rounds with 4 panellists (more rounds as a measure to improve the precision), the original personnel measuring costs increase with 50%. This comes down to 12,11 Euros extra: 9,45 Euro extra (in total) for the 4 panellists measuring one extra round and 2,66 Euro extra for the operator attending one extra round.

As a consequence:

- Measuring one sample with 5 panellists and performing only 2 rounds thus is less costly than performing 3 rounds with 4 panellists. This is because only one extra panellist needs to perform two additional rounds in the first case, while otherwise all 4 panellists need to perform an extra round, which comes down to the extra cost of 4 additional rounds.
- It also appeared that performing 2 rounds with 6 to 8 panellists is less expensive than when performing 3 rounds with 5 panellists (6 to 24% less: 2.96 up to 12.41 Euros difference per sample).

An extra reason for using a larger panel instead of performing more rounds could be that the inter-panellist variability is much smaller than the intra-panellist variability (chapter 4). In chapter 4, it was found that only 13 % of the total variation in odour thresholds could be explained by inter-panellist variability while 68 % of the total variation could be explained by the intra-panellist variability. This could also explain less outfall of panellists during retrospective screening at larger panel sizes (Chapter 5) and thereby a higher success rate of measurements when starting at a panel size higher than 4. An invalid result at panel size 4 after retrospective screening, would anyway require additional measurements by one or more extra panellists, but could be practically difficult to arrange if not foreseen in advance.
Considering that only about 40 % (Chapter 3) of the tested panel candidates passes the panel selection procedure with n-butanol, it would be more costly to organise extra panel selection tests to attain a more stringently selected panel than to perform measurements with a larger panel size or to perform more rounds.

As explained in chapter 1, dynamic olfactometry is used for the development and evaluation of abatement techniques. It is important for end users of abatement techniques that the efficiency of these techniques can be precisely quantified. This is important to consider, because it was reported in literature that measurement errors remain too large to detect relatively small effects of abatement techniques, in spite of recent improvements in dynamic olfactometry (Ubeda et al., 2013). In that view, the influence of the studied precision enhancing measures (Chapter 5) is investigated here on efficiency evaluations of abatement techniques.

The efficiency (eff) is calculated from both, the measured ingoing [C_{in}] and outgoing [C_{out}] odour concentration, using the following equation:

$$eff(\%) = \left[\frac{Cin - Cout}{Cin}\right] . 100 \qquad \text{(Eq 6.1)}$$

Therefore the precision of the calculated efficiency depends on the precision of the measurements of C_{in} as well as of C_{out}.

Specifically, the influence of different panel sizes, the number of dilution rounds (d) and the number of pairwise samples (n) on the precision of efficiency evaluations of odour abatement techniques is studied here.

Annex G of CEN (2003) (Eq 6.1) was applied to calculate 95 % confidence intervals (C.I.) on the inlet and outlet concentration of an abatement technique, considering different inlet concentrations (625 ou_E/m^3, 1250 ou_E/m^3, 2500 ou_E/m^3, 5000 ou_E/m^3 and 10000 ou_E/m^3), which are in the range of concentrations measured in pig houses (Van Langenhove & Defoer, 2002), and different reduction efficiency levels (from 10 % to 90 %) and using repeatability standard deviations (s_r), corresponding with different measurement conditions (panel sizes: 4 to 8 and 2 to 3 rounds) (Table 6.1). An example of the calculation of the confidence interval on a measured concentration can be found in appendix C. The s_r – values (Table 6.1) for different measurement conditions are deviated from the precision's study (Chapter 5) considering pig odour measurements of ILVO's laboratory.

$$\overline{y_w} - t \cdot \frac{s_r}{\sqrt{n}} \leq m \leq \overline{y_w} + t \cdot \frac{s_r}{\sqrt{n}} \qquad \text{(Eq 6.1)}$$

$\overline{y_w}$: average of the test results

t : student f factor (t = 2 considering a 95% C.I.)

m: expected value of test results

s_r: repeatability standard deviation

n: number of replicate samples

Table 6.1 Repeatability standard deviations for the ILVO laboratory calculated for pig odour (Chapter 5)

Panel size	<s_r> pig house odour	
	2d	3d
4	0,11	0,10
5	0,10	0,09
6	0,09	0,08
7	0,08	0,07
8	0,07	0,06

The purpose was to assess the minimum requirements (Table 6.3) to evaluate reduction efficiencies of abatement techniques with dynamic olfactometry.

When the respective C.I. on the inlet and on the outlet concentration did not overlap, the concentration at the inlet could be considered significantly different from the

concentration at the outlet of the abatement technique. The corresponding conditions were assumed applicable for evaluating the efficiency of the abatement technique. The minimum requirements were then defined to be the minimum measurement conditions to achieve a significant difference between in- and outlet. This principle is illustrated in table 6.2 for an abatement technique with an inlet concentration of 10000 ou_E/m^3, an efficiency of 40 % and n = 3 pairwise samplings. The minimum requirements in that situation are indicated in green (Table 6.2).

Table 6.2 Respective C.I for an abatement technique with an inlet concentration of 10000 ou_E/m^3, an efficiency of 40 % and n = 3 pairwise samplings. Determination of the minimum requirements (in green) for measuring a significant difference between in- and outlet.

Panel size	N rounds	Confidence interval on odour concentration at inlet [ou_E/m^3]		Confidence interval on odour concentration at outlet [ou_E/m^3]	
		UnderLimit	UpperLimit	UnderLimit	UpperLimit
4	2d	7516	13304	4510	7983
	3d	7709	12971	4626	7783
5	2d	7672	13034	4603	7820
	3d	7850	12738	4710	7643
6	2d	7891	12673	4734	7604
	3d	8085	12369	4851	7421
7	2d	8048	12425	4829	7455
	3d	8232	12148	4939	7289
8	2d	8260	12107	4956	7264
	3d	8430	11863	5058	7118

The sr (Table 6.1) were also used to calculate the C.I. on the estimated reduction efficiency (Table 6.3) for the different minimum requirements and this according to Annex H of CEN (2003) (Eq 6.2). Logarithmic transformation of the concentrations is required.

$$\overline{y_D} - t \,.\frac{s_D}{\sqrt{n}} \leq m_D \leq \overline{y_D} + t \,.\frac{s_D}{\sqrt{n}} \qquad \text{(Eq 6.2)}$$

$\overline{y_D}$: average of the test results

t : student t factor (t = 2 considering a 95% C.I.)

m_D : expected value of the difference between the test results before and after the aircleaning treatment

s_D: variance of the difference between the test results before and after the aircleaning treatment

n: number of replicate samples

$s_D^2 = 2.s_r^2$ or $s_D = \sqrt{2} \,.\, s_r$

As a result, table 6.3 shows the minimum requirements in panel size and in number of rounds established for measuring a significant difference (with 95 % confidence) between respectively the odour concentration at the inlet and the odour concentration at the outlet of the odour abatement technique. Table 6.3 also presents the 95 % - confidence intervals (C.I.) on the assumed reduction-efficiencies based on the corresponding measurement conditions. The number of samples (n) taken both at the inlet and at the outlet of the odour abatement technique are also represented (Table 6.3).

From table 6.3 can be deduced that the in- and outlet concentration of an odour abatement technique with an estimated reduction efficiency of **60 % to 90 %** and for a starting concentration ranging from 625 to 10000 ou_E/m^3 can be distinguished with 95 % confidence when using at least a panel size of 4 and 2 rounds. This applies both when 2 or 3 samples are taken at the in- and outlet of the abatement technique (Table 6.3).
For the same inlet concentration range, but performing 3 samplings at the in- and outlet of the abatement technique, the inlet and outlet concentration of a **50 %** reduction technique will also be distinguishable when measuring with 4 panellists and performing 2 rounds (Table 6.3). When only 2 samples are taken at both the inlet and at the outlet of the technique, the inlet and outlet concentration of a 50 % reduction technique will be distinguishable with 4 panellists and 3 rounds (Table 6.3).

Table 6.3 Minimum requirements for the evaluation of odour abatement techniques based on the distinctiveness of the C.I. on the respective inlet and outlet concentration

inlet conc. ($ou_E\ m^{-3}$) [a]	Est. efficiency (%) [b]	n [c]	Min. panel size [d]	Min. rounds [e]	C.I. on efficiency [f]
625 up to 10000	90	2	4	2	83,6 % ≤ 90,0 % ≤ 93,9 %
		3	4	2	85,0 % ≤ 90,0 % ≤ 93,3 %
	80	2	4	2	67,2 % ≤ 80,0 % ≤ 87,8 %
		3	4	2	70,1 % ≤ 80,0 % ≤ 86,6 %
	70	2	4	2	50,8 % ≤ 70,0 % ≤ 81,7 %
		3	4	2	55,1 % ≤ 70,0 % ≤ 80,0 %
	60	2	4	2	34,4 % ≤ 60,0 % ≤ 75,6 %
		3	4	2	40,1 % ≤ 60,0 % ≤ 73,3 %
	50	2	4	3	21,5 % ≤ 50,0 % ≤ 68,1 %
		3	4	2	25,1 % ≤ 50,0 % ≤ 66,6 %
	40	2	7	3	16,0 % ≤ 40,0 % ≤ 57,2 %
		3	5	3	15,5 % ≤ 40,0 % ≤ 57,4 %
	30	2	/	/	/
		3	8	3	10,9 % ≤ 30,0 % ≤ 45,0 %
	20	2	/	/	/
		3	/	/	/

[a] Odour concentration present at the inlet of the abatement technique
[b] Estimated odour reduction efficiency (%)
[c] The number of samplings at the inlet and at the outlet of the abatement technique
[d] Minimum panel size
[e] Minimum number of rounds
[f] The corresponding confidence interval on the estimated reduction efficiency
/ : Even with 8 panellists and performing 3 rounds in these conditions, there is an overlap between the 95%-confidence interval on the inlet concentration and the 95%-confidence interval on the outlet concentration.

Considering a **40 %** odour reduction technique, a panel size of 5 and performing 3 rounds is the minimum requirement to be able to adequately assess a reduction efficiency when applying 3 pairwise samplings (Table 6.3). If only 2 samples are taken at both the inlet and at the outlet of the abatement technique, a minimum panel size of 7 and performing 3 rounds are required to measure a difference between the in- and outlet-concentration of a 40 % odour abatement technique (Table 6.3). This applies for example when evaluating a biological air scrubber with an assumed efficiency of 40 % (Vlaamse overheid & Leefmilieu Natuur en Energie, 2016).
The in- and outlet-concentration of a **30 %** odour reduction technique will only be significantly different, when performing 3 samplings on the in- and outlet of the technique and when measuring with 8 panellists and performing 3 rounds (Table 6.3). Taking 2 samples at the inlet and outlet, the inlet and outlet concentration of a 30 % odour reduction technique (e.g. a chemical air scrubber, ref. (Vlaamse overheid & Leefmilieu Natuur en Energie, 2016) would not be statistically distinguishable considering the tested inlet concentration range (625 – 10000 ou_E/m^3) and the tested measuring variables (panel sizes 4 to 8; performing 2 or 3 rounds) (Table 6.3). In that case more pairwise samplings and measurements would be necessary.
The inlet and outlet concentration of techniques with an efficiency of **20 %** or **10 %** cannot be distinguished in any of the tested conditions (Table 6.3). A minimum of 8 pairwise samplings, panel size 8 and 3 rounds would be needed to be able to significantly distinguish in and out with 95 % certainty from a 20 % reduction technique.

It should be noted that the s_r (Table 6.1), used in this assessment, were much smaller than the minimum requirement of CEN ($s_r \leq 0.1721$). The results (Table 6.3) thus suggest that even when measuring at a higher precision than CEN prescribes (s_r in the order of 0.064 - 0.107 (Table 6.1), versus a maximum s_r of 0.172 (CEN-requirement)), the minimum requirements to be able to measure a significant difference between in- and outlet of a 30 % - 40 % odour reduction technique are higher than the minimum requirements set by CEN for a single measurement (panel size 4; 2 rounds). It should be noted however that CEN indicates to choose the number of samplings as a function of the desired precision (Annex G and H of CEN (2003)).

The results also suggest that even when measuring with a higher precision than required by CEN (2003), the reduction effect of source oriented techniques and of windshields, which have estimated reduction efficiencies in the order of 10 – 25 % (Vlaamse overheid & Leefmilieu Natuur en Energie, 2012), cannot be detected by dynamic olfactometry, even when starting at panel size 8, performing 3 rounds and taking 3 samples at the inlet

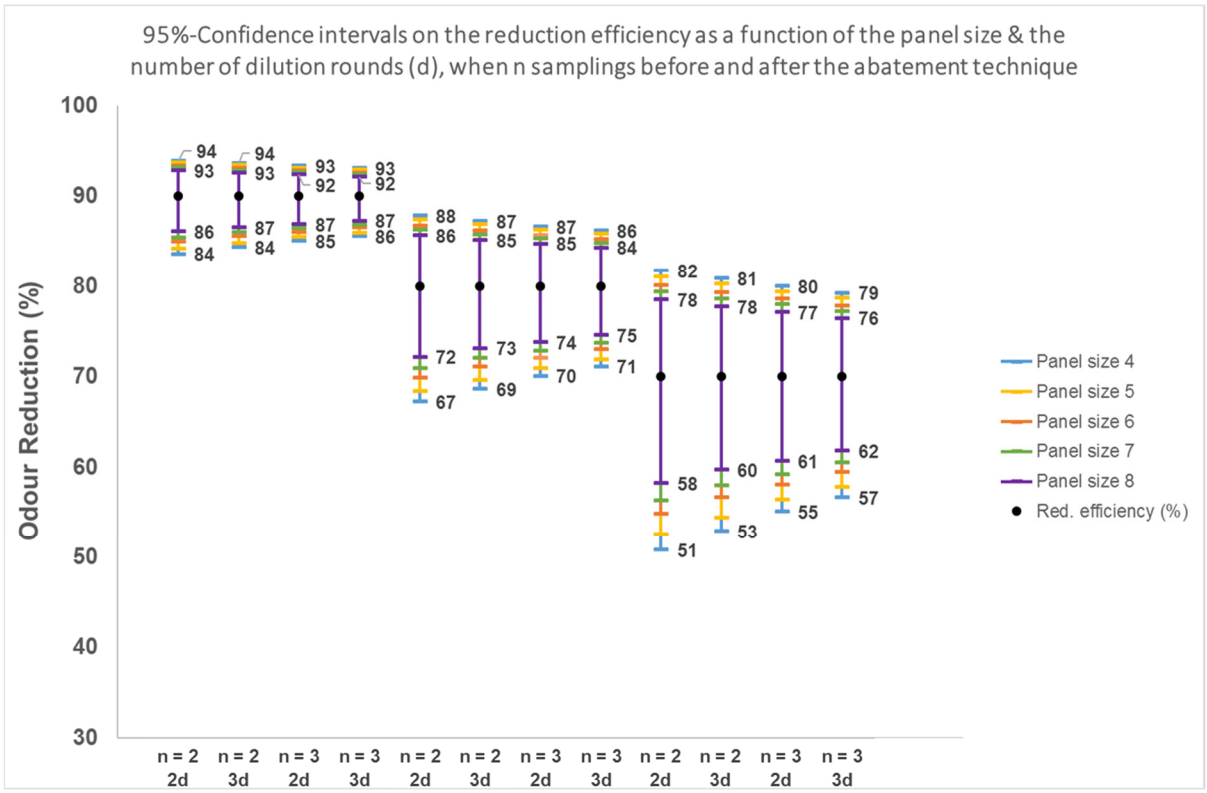
and outlet. This is in line with the results of Chapter 3 (Diksmuide-campaign) where no significant difference in odour concentration could be found between traditional and low ammonia emission compartments. Also no significant difference was found between the odour concentration before and after cleaning the compartments. In those circumstances, complementary chemical analyses could be used to assess the reduction of important odorants.

Table 6.3 also shows that the width of the confidence intervals at the minimum requirement for reduction efficiencies of 90 %, 80 %, 70 %, 60 % and 50 % is equivalent to their respective complement to make up to 100 % (e.g. C.I. for 60 % efficiency has a width of about 40 %). For a reduction efficiency of 40 %, the confidence interval at the minimum requirement (panel size 5 and 3 rounds for n = 3 or panel size 7 and 3 rounds for n = 2) has nearly the same size as the effective reduction efficiency of the technique (so +/- 40 %). For a 30 % odour efficiency the confidence interval at the minimum requirements has a width of about 30 %. Since the confidence intervals are relatively wide at the minimum requirements (Table 6.3), the exact value of the measured reduction efficiency is quite uncertain. From these findings, it is thus preferable to start from higher conditions than the minimum requirements to be able to measure the reduction efficiency more accurately, generating a lower spread on the result and thus a smaller confidence interval. Yet, as explained before, the minimum requirements are only to ensure to measure a difference with 95 % certainty between in and out. For a more accurate estimation of the reduction efficiency, higher start conditions will be needed. It should be noted however that the confidence intervals presented here, were calculated at higher precision circumstances (Table 6.1) than the minimum requirements set by CEN (2003) ($s_r \leq 0.1721$).

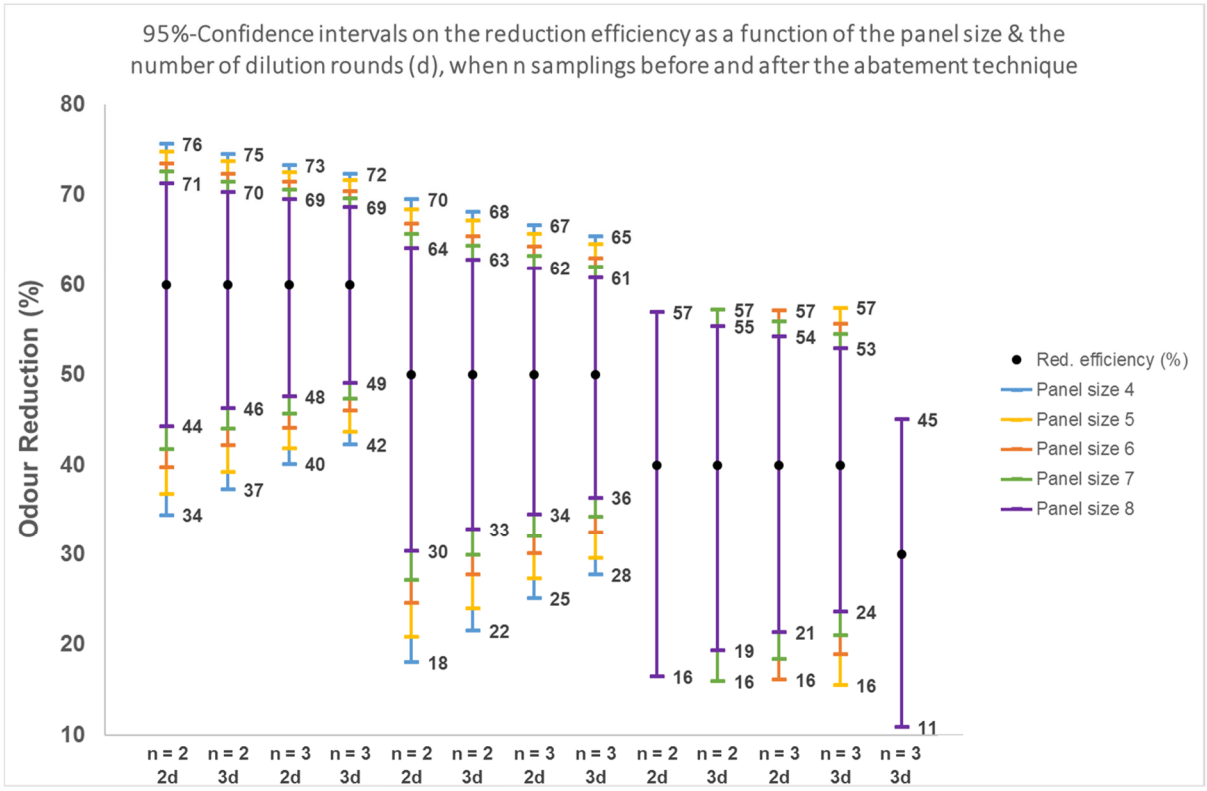
Figure 6.1 shows to what extent the assessment of reduction efficiencies can be further improved compared to the minimum requirements (Table 6.3). Therefore, the confidence intervals (C.I.) were calculated according to annex H of CEN (2003) (Eq 6.2) on the reduction efficiencies of 30 – 90 % for panel sizes of 4 to 8 and for both 2 and 3 rounds of dilutions (2d and 3d), considering the corresponding s_r, (Table 6.2) and as well for n = 2 as for n = 3 samplings at the inlet and outlet of the reduction technique. These C.I. were calculated independent of the inlet concentration of the reduction-technique following annex H of CEN (2003). The C.I. are displayed in Figure 6.1, starting from the minimum requirements to be able to measure a difference with 95 % confidence (Table 6.1) and ending with the maximum assessed conditions (panel size of 8, 3 rounds, n = 3).

From figure 6.1 can be deduced that the C.I. on the reduction efficiencies reduce with an increasing panel size, an increasing number of rounds and an increasing number of samples. Different panel sizes are indicated by a different colour (Fig. 6.1a).
It can also be seen that the C.I. for reduction efficiencies of 70 % up to 90 % (Fig. 6.1a) are much smaller than the C.I. for reduction efficiencies of 30 to 60 % (Fig 6.1b). Reduction efficiencies of 70 % up to 90 % are usually found for combined air scrubbers (Van der Heyden et al., 2015). Figure 6.1 also shows that:

- For a 90 % reduction efficiency (Fig 6.1, Appendix B), the C.I. is rather small already at panel size 4 and 2d and n = 2. The width of the C.I. for 80 % efficiency can nearly be halved when moving from panel size 4 towards panel size 8 for n = 2 and 2d (Fig 6.1, Appendix B). Performing 3d or n = 3 samplings has less extra value than enlarging the panel. For 70 % efficiency the C.I. can be reduced by nearly one third moving from the minimum requirements (Table 6.1) towards panel size 8 when performing 2d and n = 2 (Fig 6.1, Appendix B). Performing 3d or n = 3 has less extra value than enlarging the panel (Fig 6.1, Appendix B).
- Applying precision enhancing measures is advisable when evaluating the abatement effect of a technique with an estimated efficiency of 50 to 60 %, since the C.I. at the minimum requirements is rather large when evaulating these efficiencies (Fig. 6.1). An efficiency of 50 to 60 % could for example be measured for reducing the surface contact between slurry and air (Ubeda et al., 2013).
- Measuring a reduction efficiency of 40 % and 30 % requires already a higher measuring effort to be able to significantly distinguish in and out concentration of the reduction technique: a panel size of 7 is the minimum for evaluating an efficiency of 40 % when performing 3d and n = 2 and a panel size of 5 in case of 3d and n = 3 (Table 6.1). A technique with 30 % efficiency can only be assessed with panel size 8, n = 3 and 3d.

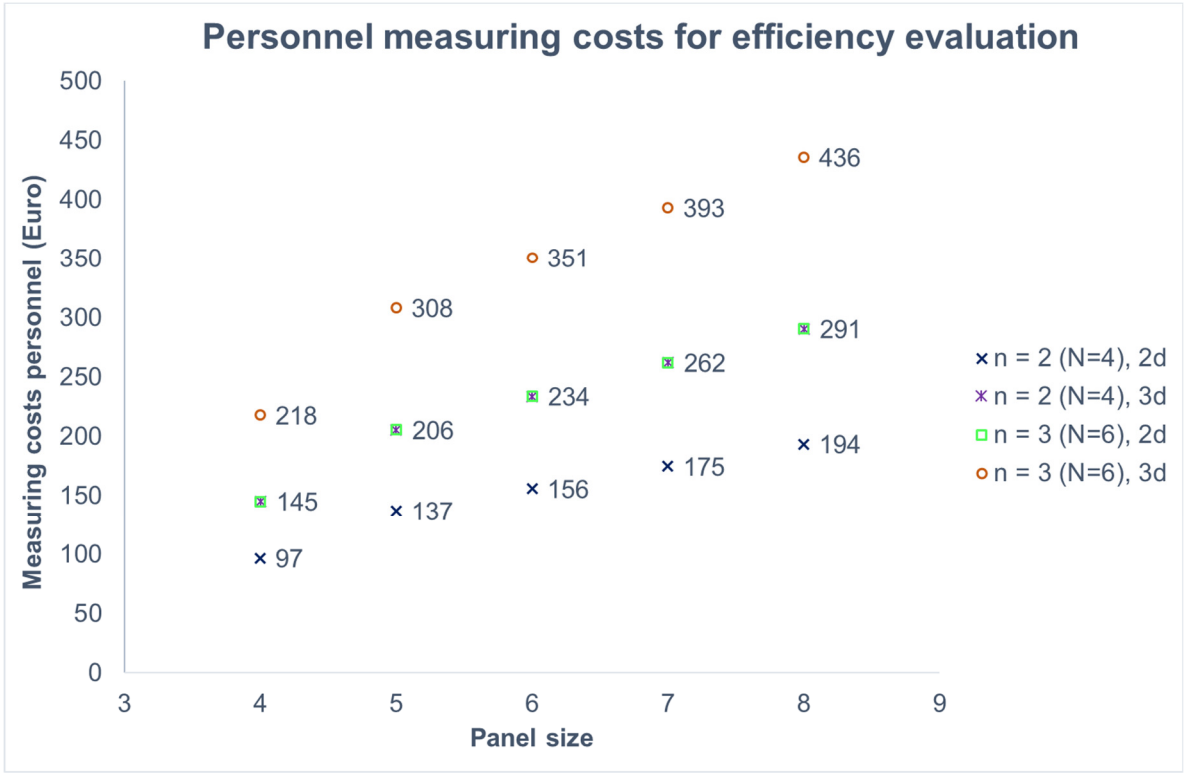


(a)

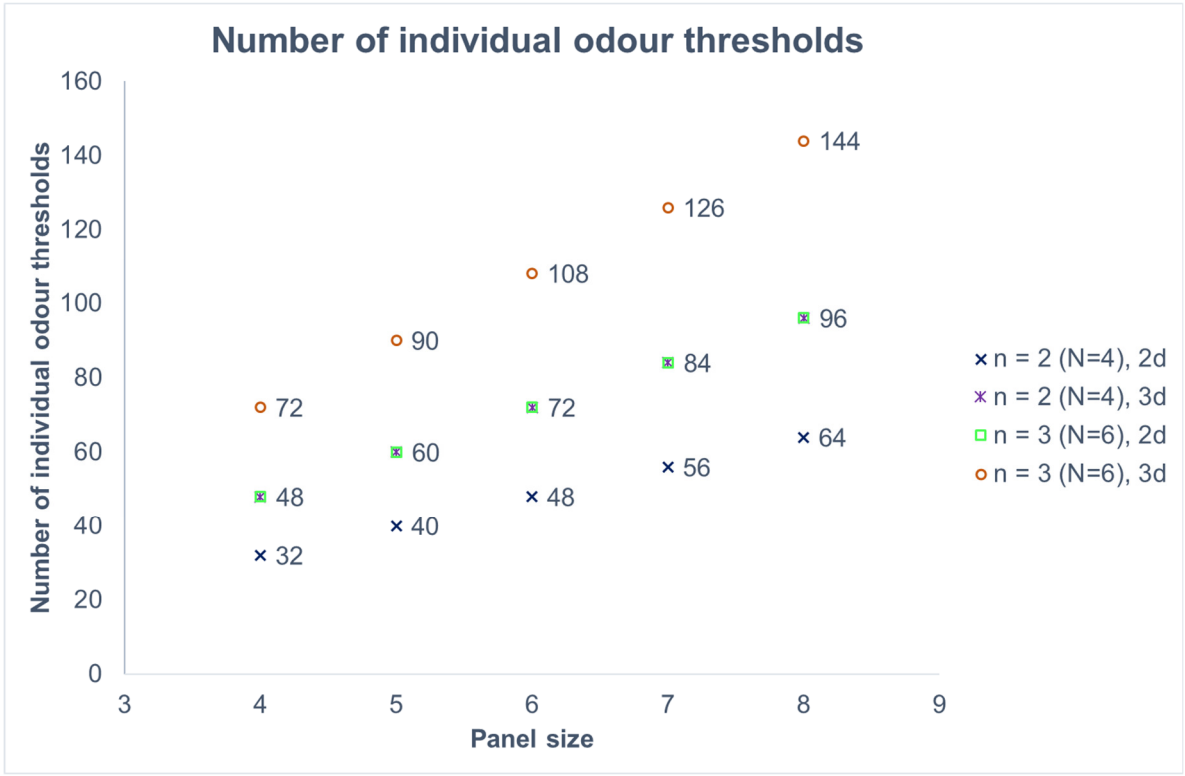


(b)

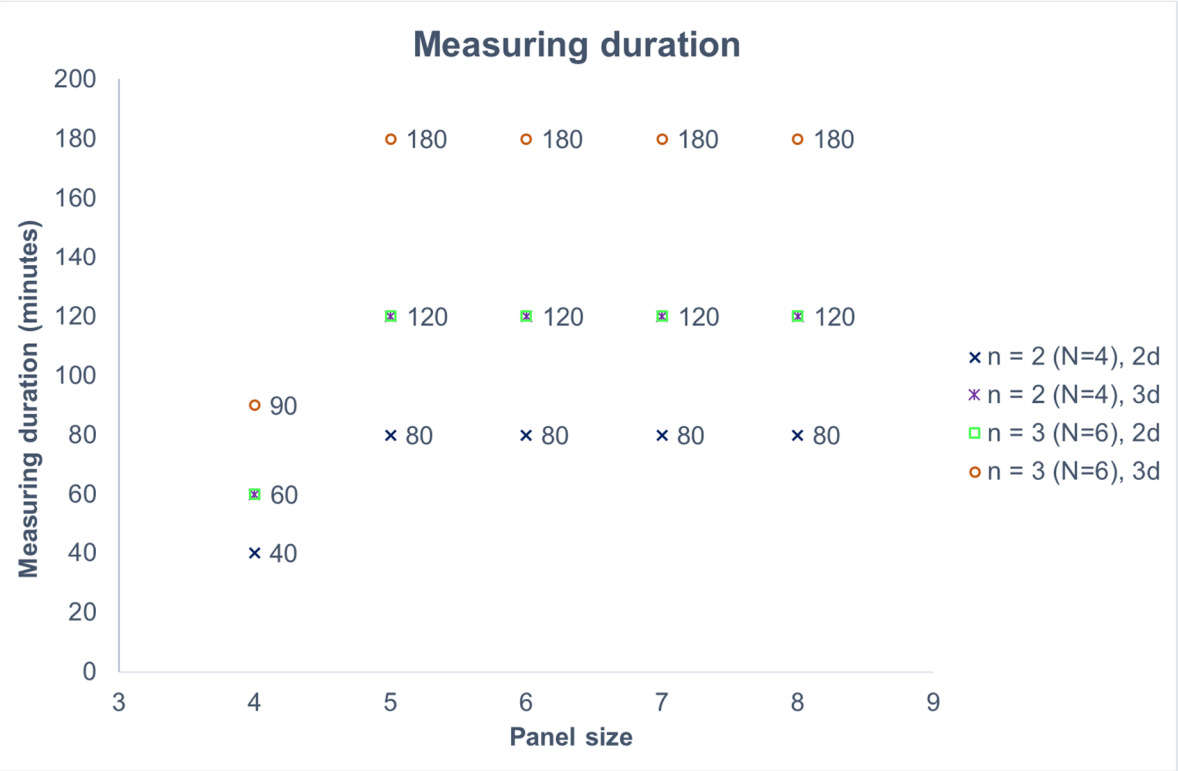
Figure 6.1 (a): 95 % confidence intervals on assumed reduction efficiencies of 70 % to 90 % for different measuring conditions (b) 95 % confidence intervals on assumed reduction efficiencies of 30 % to 60 % for different measuring conditions


Personnel measuring costs for efficiency evaluation
Measuring costs personnel (Euro)
500
450
400
350
300
250
200
150
100
50
0
3
4
5
6
7
8
9
Panel size
436
393
351
308
291
262
234
218
206
194
175
156
145
137
97
n = 2 (N=4), 2d
n = 2 (N=4), 3d
n = 3 (N=6), 2d
n = 3 (N=6), 3d

(a)


Number of individual odour thresholds
Number of individual odour thresholds
160
140
120
100
80
60
40
20
0
3
4
5
6
7
8
9
Panel size
144
126
108
96
90
84
72
72
64
60
56
48
48
40
32
n = 2 (N=4), 2d
n = 2 (N=4), 3d
n = 3 (N=6), 2d
n = 3 (N=6), 3d

(b)



(c)

Figure 6.2 (a) Personnel measuring costs for different measuring conditions (b) Corresponding number of individual thresholds (c) Total measuring duration

As Gostelow et al. (2001) mentioned, attaining a balance between resolution and costs is important when performing olfactometry.

In that perspective, figure 6.2 presents the estimated costs, the assessed number of individual odour thresholds and the measuring duration according to the different measurement conditions. N represents the total number of samples measured for the evaluation of an abatement technique and is equal to 2 times n (e.g. n = 2 samples at each side (in- and outlet) of the technique, gives N = 4 samples in total).

- Figure 6.2a shows that using 5 panellists and 2 rounds (2d) is less costly than using 4 panellists and performing 3 rounds (3d) for N = 4 (thus n = 2 samplings per side of the abatement technique), while the precision achieved is comparable (Fig 6.1, appendix B). Using 6, 7 or 8 panellists and performing 2 rounds is less costly than using 5 panellists and 3 rounds for N = 4 (Fig 6.2a), while a better precision could be achieved with a higher panel size (Fig 6.1, appendix B).
- Considering the same panel size, measuring 4 samples and each in 3 rounds (N=4, 3d) is as expensive as measuring 6 samples and each in 2 rounds (N=6, 2d) (Fig 6.2a), but according to figure 6.1 more samples can generate a higher precision compared to more rounds, so if practically possible, performing more samplings would be preferable to more rounds.

- For 60 % efficiency, when moving from panel size 4, n=2 (N=4), 2d (minimum requirement) towards panel size 4, n=3 (N=6), 2d; the 95 % C.I. could reduce in width from [34 ≤ 60 ≤ 76] towards [40 ≤ 60 ≤ 73] with an additional cost of 48 Euros (over in total 6 samples) (Fig 6.1 and 6.2, Appendix B). It appears that the largest effect in precision with the smallest additional cost could be achieved by moving towards panel size 8, n=2, 2d (Appendix B).
- For 50 % efficiency, when moving from panel size 4, n=2 (N=4), 3d (minimum requirement) towards panel size 4, n=3 (N=6), 2d; the 95 % C.I. could reduce in width without an additional cost. When moving towards panel size 8, n=2 (N=4), 2d the 95 % C.I. could reduce in width from [22 ≤ 50 ≤ 68] towards [30 ≤ 50 ≤ 64] with an additional cost of 48 Euros (over in total 6 samples). This is the largest effect in precision with the smallest additional cost (Appendix B).
- Measuring 4 samples (2 pairwise samplings over in- and outlet) with 8 panellists in two rounds is less expensive (40 Euros difference, Fig 6.2) and more precise (Fig 6.1) than measuring 4 samples with 6 panellists in 3 rounds and thus the first scenario could be preferred.

It should be noted that when performing olfactometric measurements in practice for the evaluation of abatement techniques and considering that the efficiency of abatement techniques varies in time, it will be necessary also to perform measurements at different time-intervals. However, the purpose of the above mentioned calculations was to derive the uncertainty on an efficiency evaluation at a given time within the assessment period.

6.2 Representativeness of the reference gas and implications for practice

The differences found between n-butanol and pig house odour during the performed odour measurements (Chapter 4) and within the performed precision study (Chapter 5) rise questions which complement the ongoing debate about the representativeness of the reference odour (Laor et al., 2014; Parker et al., 2005; Klarenbeek et al., 2014). Based on the measuring and simulation results and within the set-up of this PhD-study, the sensitivity of individual panellists towards n-butanol did not seem to predict their individual pig house odour sensitivity (Chapter 4) and a significantly better precision was found for pig odour compared to n-butanol (Chapter 5).

The differences between the performance for pig odour and n-butanol are probably related to the physiology of the human sense of smell, where more olfactory receptors

are stimulated by a complex odorants' mixture such as pig odour compared to a singular substance such as n-butanol. Only a few receptors of the human nose can detect n-butanol, while an odour mixture can be perceived by a lot of olfactory receptors (www.olores.org, 2015b) Pig odour could therefore be more clearly detected by human panellists than n-butanol, leading to more uniform or repeatable results among panellists for pig odour (compared to n-butanol).

Differences in performance between n-butanol and environmental odours (odour mixtures) were also found in previous researches, namely by Parker et al. (2005) and McGinley & McGinley (2010) concerning the predictability assumption of CEN (2003) (Chapter 4) and also by Laska & Hudson (1991); Sneath (2003) and Van Harreveld & Heeres (1995), when comparing the precision for both odours. Zernecke et al. (2011), who found significant differences in sensitivity between different odorants (e.g. comparing the sensitivity to n-butanol and isobutanol with the sensitivity to both phenylethyl alcohol and isoamyl butyrate), explained that these differences could be due to differences in structure and in functional groups of the respective odorants, which on their turn elicit activity from different receptors. These differences appeared when measuring with normally sensitive persons (normosmic), which are generally pursued during the panel selection process. The Task Group dealing with the calculation of uncertainty in the revision process of CEN (2003) (CEN/TC264/WG7) recently approved that n-butanol is probably not representative for all environmental odours and that this reference gas does not necessarily represent the sense of smell of a panellist very well (www.olores.org, 2015b). According to this source, a malfunctioning of one of the few receptors that can perceive n-butanol could largely influence the panellist's perception of n-butanol, while not having a large effect on this panellist's overall sense of smell (www.olores.org, 2015b). It should be noted also that olfactory receptor cells (neurons) are constantly renewed: in mice this happens every 30 days (Schamp & Van Langenhove, 1987; Moulton, 1975), which could also influence the olfactory perception.

The fact that n-butanol might not be representative for all environmental odours poses problems in practice:

- This makes the traceability of odour concentrations questionable: panellists are now selected according to their performance towards n-butanol, under the assumption that the sensitivity for the reference predicts their sensitivity to other substances and this to ensure the traceability of the odour concentration of any odour towards the reference odour. So, it is possible that the panel selected by the current selection procedure with n-butanol (CEN, 2003) might not be average sensitive towards environmental odours.

- It is possible that the panel selection process with n-butanol might be too severe (only a few of the noses' receptors can detect n-butanol) and so potential average sensitive persons towards environmental odours might be excluded. As mentioned before, the success rate of panel selection with n-butanol is generally very low: literature reports that only 30 - 50 % of the tested assessors pass the panel selection procedure (Munoz et al., 2010), which increases the efforts for panellists' selection.

As already stated by CEN (2003), an odour mixture as a reference would be preferable. Clanton et al. (1999) suggested to replace n-butanol by a reference odour similar to the odour under survey to reduce the panellist variation and thereby improve the precision of odour measurements. Klarenbeek et al. (2014) proposed to use a reference gas that reflects the characteristics of waste gases that are usually found in practice.
Searching for alternative reference gasses seems opportune. An alternative reference gas should have the following characteristics. Preferably, it would be an odour mixture (CEN, 2003), which is chemically stable (Qu & Feddes, 2007) and its compounds should additively interact (the odour threshold for the mixture will then rise directly proportional to the increase in the concentration of its compounds), so no antagonistic or synergetic effects between its compounds. The reference gas should be easy to manufacture and to handle and be safe in use for the human panellists (Qu & Feddes, 2007). It should not contaminate the olfactometer, but preferably have an odour character similar to the odour to be measured (Qu & Feddes, 2007). This could lead to the use of multiple reference gases depending on the odour type. For instance, H_2S (hydrogen sulphide) is used in Germany as an additional reference gas (Mannebeck & Mannebeck, 2001). McGinley & McGinley (2010) also found H_2S useful to include in the panel selection procedure. Also THT (tetrahydrothiophene) is used in Germany during interlaboratory comparison tests. In Japan, multiple standard odours are used (Saiki, 2003), namely β-phenylethyl alcohol, methyl cyclopentanolone, γ-undecalactone, isovaleric acid, and skatole, of which the last two have an unpleasant smell. When searching for an alternative reference odour, the knowledge gained from previous studies should be taken in mind: Qu and Feddes (2007) developed an artificial swine odour based on fatty acid, reduced sulphur, aliphatic alcohol, aliphatic hydrocarbon, indole and aromatic alcohol compounds. In the study by Defoer and Van Langenhove (2002), the use of a synthetic gas mixture composed of ethane thiol, methylacetate and 2-propanol in nitrogen was studied.
In the revised CEN (2003) standard, which will be an ISO-standard, a procedure will be foreseen to test other odorants for use as a reference odorant, but it should elicit a physiological response equivalent to that of n-butanol (Van Harreveld, 2014). It is however questionable whether an odour mixture could elicit a similar physiological

response as a singular substance such as n-butanol. Possibly this additional test procedure will lead to additional reference gasses in the form of singular substances, for example H_2S or THT. H_2S or THT already have an odour character (unpleasant odour) more similar to pig odour, but require handling with care to ensure the panellists´ safety as they are toxic and irritating at relatively high concentrations.

6.3 Benefit of performing odour simulations

Using the simulation tool, described in chapter 5, 40500 odour concentrations could be generated in a short period of time. Considering an average measuring time of 15 min. per sample, the simulation tool allowed to save up 10125 analyses' hours. The developed tool could be used for precision analysis on larger datasets. Eventually, the performed assessments could be repeated using other odour types (e.g. from poultry houses or even samples from industrial sources) to gain more insight in the precision of olfactometry assessments. The effect of for example 4 rounds was not included in this thesis, but could be tested also in combination with different panel sizes. Repeating the simulations on datasets of odour laboratories could inform if the investigated measures also apply for other laboratories or whether there are large interlaboratory differences at this level also. It would allow to draw conclusions based on existing datasets from other laboratories. Performing odour simulations was encouraged by Ogink et al. (1995) and Boeker et al. (2008), since olfactometry practice is labour-intensive and time-consuming.

6.4 Olfactometry and other odour measurement methods

The use of olfactometry in odour studies has the advantage that it directly uses the human nose, which is a broad spectrum and very sensitive odour detector (Brattoli et al., 2011) and that it measures the perception by humans.

When olfactometry is used to evaluate the reduction efficiency of an abatement technique and the estimated efficiency of the technique is rather small (e.g. 30 % or lower), it is however advisable to use complementary odour measuring techniques. Chemical analysis can be used to measure small changes in concentrations of specific malodour compounds (Van Huffel et al., 2012; Bruneel et al., 2016; Hansen et al., 2014) and this with a high accuracy and precision (Gostelow et al., 2001; Munoz et al., 2010). Chemical analyses also give an insight in the chemical processes responsible for the odour production and also in the odour reduction processes induced by abatement

techniques. Chemical analysis thus can be considered complementary to olfactometry when developing or evaluating abatement techniques.

Different experiments have been performed to study the correlation of results from chemical analysis and from dynamic olfactometry, but led to different outcomes. Kim and Park (2008) found that the odorants' concentration data of offensive odours could be used for odour intensity estimations of industrial air samples, but that it was difficult to predict whether this would apply for samples with lower odour concentrations (less polluted samples). Hansen et al. (2016) could explain 74 % of the variation in odour concentration, determined by field olfactometry, by chemical analysis of odorants with PTR—MS. Trabue et al. (2011) found few significant correlations (only with ammonia and hydrogen sulphide) between the chemical concentration of odorants and the dilution thresholds measured by dynamic olfactometry. According to them applying dynamic olfactometry for odour quantification of swine operations is challenging because of the nature of agricultural odours (diverse and polar, adsorbing on storage containers, transforming before analysis) and a possible low correlation between the analytical standard (n-butanol) and odorous compounds from animal production, particularly in their ability to be diluted. Currently odour activity values (OAV) of individual compounds (i.e. the ratio of an odorant's concentration in air to its odour threshold) are being used in correlation analyses (Trabue et al., 2011, Blazy et al., 2015).

The sniffing team method (Bilsen et al., 2008; EN 16841) has both the advantage that it includes the human nose as a sensor and that the odour is directly measured in the field after being naturally diluted by its existing environment (instead of mechanically diluted by the olfactometer) and thus directly measures the odour perceived by neighbouring residents. The advantage of this method is that its' working principle is more closely related to the functioning of the human sense of smell, namely that the human nose is more adapted to detect changes in odour concentrations rather than statically determine the absence or presence of an odour (working principle of dynamic olfactometry). The European standard for the sniffing team method (EN16841) has been ratified, unfolding two methods for the direct assessment of odour in ambient air (Van Elst & Delva, 2016; www.olores.org, 2015a). A recent study (Driesen et al., 2016) promotes the use of the sniffing team method to allow the individual evaluation of the odour dispersion of a farm. Sociological methods (Driesen et al., 2016; Van Broeck & Van Langenhove, 2000) on their turn have the advantage to directly monitor the nuisance effects in the surrounding of a pig farm.

So far, the dose – response relationship of livestock emissions and the resulting perception or odour nuisance is not quantified yet. So, integrating all odour measurement techniques (olfactometry, sniffing team method, chemical and sociological analysis),

which could be considered as being complementary (Munoz et al., 2010), does result in the most information concerning an odour situation. Together they can inform on the odour mixture that is formed in the pig house as well as provide an understanding on the dilution of the odour in the environment. The odour concentration that attends the neighbouring houses could be assessed and the effect the odour emission has (nuisance or not) on the neighbours. Together they can also inform on the effectiveness of odour abatement techniques.

CHAPTER 7

GENERAL CONCLUSIONS

CHAPTER 7: GENERAL CONCLUSIONS

In this thesis, the use of dynamic olfactometry, according to CEN (2003), to objectively measure odour concentrations and emissions, generated by pig houses has been evaluated. This research started with a critical literature review on dynamic olfactometry and the development of an odour laboratory at ILVO, specifically dedicated to pig house odour measurements.

Different methodological issues associated with the application of dynamic olfactometry according to CEN (2003) for the measurement of pig house odour were defined. The main issues were the large measuring uncertainty associated with dynamic olfactometry and the questioned representativeness of the reference gas n-butanol.

Also some practical issues that can occur when establishing an olfactometric laboratory according to CEN (2003) were addressed, namely the large drop-out of odour panel candidates during panellists' selection with n-butanol and the elimination of qualified panellists when applying the retrospective screening procedure of CEN (2003) during odour measurements.

The main research results are presented in figure 7.1. In summary, based on the performed, comparative n-butanol and pig house odour measurements the n-butanol sensitivity of panellists did not seem to predict their pig house odour sensitivity. Also a significant difference in precision was found between pig house odour and n-butanol, causing to question the transferability assumption of CEN (2003), regarding pig house odour.

All measures, investigated with the purpose of improving the precision, significantly enhanced the precision of the odour measurements, namely a higher panel size, a higher number of dilutions' rounds and a higher individual repeatability of the panellists. Increasing the panel size, the number of rounds and the number of samplings could also decrease the uncertainty on efficiency evaluations of abatement techniques.

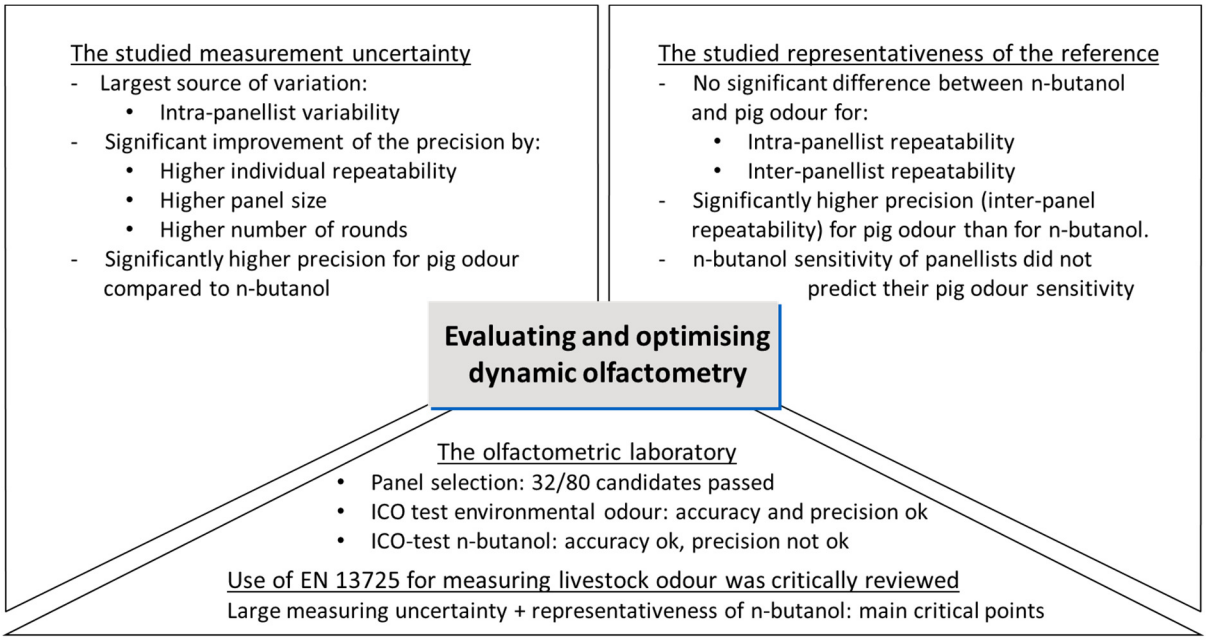


Figure 7.1 Main research results

Based on the research results the following recommendations for future research could be formulated:

- In this study, the influence of replicate samples, the use of different panels, panellists, and the individual variability in sensitivity of panellists on the total variation of the performed olfactometric measurements have been investigated (Chapter 4). Afterwards, the effect of the panellist's performance level, the panel size, the number of rounds and the odour type under investigation on the olfactometry precision have been studied (Chapter 5). As could be deduced from the literature review (chapter 1.8), processes such as sampling, sample storage and diluting of the odorous sample in the olfactometer affect the chemical composition of odour samples and thereby will influence the concentration that is measured by dynamic olfactometry. The effects of these processes on the measurement uncertainty should be quantified by olfactometric experiments, to increase the insight in the total measurement uncertainty related to dynamic olfactometry. In that view, further research which maps and quantifies the effect of the different sources of variation at the different stages of the olfactometric procedure is indispensable to be able to quantify the total measurement uncertainty on olfactometric measurements. To this end, it is advised to perform interlaboratory assessments to include also the variation between laboratories. Quantifying the total measurement uncertainty on dynamic olfactometry, would enable to nuance the calculated odour immissions when being compared to prevailing odour regulations. A confidence interval could for example be formulated next to the calculated odour emissions.

- Since the investigated precision improving measures proved to have a significant effect on the olfactometric precision in this study, it would be interesting for other labs to repeat the performed investigations. Odour simulations on existing datasets could support these investigations and help to reduce the laboratory efforts in these assessments.

  If the investigated measures would have a comparable effect for other laboratories, they could be a means to improve the reproducibility between labs. Since significant differences were found, when exploring the limits set by EN regarding the individual repeatability, the panel size, the number of rounds within this study, it seems that these elements could be important to consider when further harmonising measurement protocols with the purpose of improving the reproducibility between laboratories.

- Differences in performance between practice odours and the reference odour, n-butanol were found in the study by Klarenbeek et al. (2014) and during this PhD study specifically dedicated to pig house odour measurements. This indicates that further research on this topic is necessary. It would be interesting for other laboratories to repeat the investigations regarding the transferability of the reference gas, to exclude the lab-dependany of these findings.

- Since odour nuisance problems not only apply for pig houses, It would be interesting to repeat the performed investigations, including other environmental odours.

- As shown from the literature review, some alternative gasses for the reference have been tested, but no alternative reference gas is yet established. The search for an alternative for n-butanol should be continued. Ideally, an alternative reference gas would be an odour mixture (compared to the single substance, n-butanol) with properties similar to the odour type to be measured. Since dynamic olfactometry is used as well for the measurements of odours from agriculture as from industrial sources, the development of different reference gasses depending on the odour source type seems opportune. In the search for an alternative for n-butanol and in order to adequately measure pig house odours, the response towards odour mixtures including important livestock odorants could be tested and the average sensitivity of a large population's response for this odour type could e.g. be evaluated (in analogy with the suggestion of Nicolai et al., 1997).

- Since odour abatement techniques with a lower reduction potential (with efficiency of 40 % or less) appear to be more difficultly evaluated (Chapter 6) by dynamic

olfactometry, supporting these olfactometric evaluations with chemical analysis should be further continued. Chemical analysis cannot be used to measure the perception effect, but could be used to investigate the reduction of specific odour compounds, which appear to be important in the livestock odour mixture. Since the hedonic character of livestock odours can change through the use of odour reducing techniques, it would be interesting to take into account also the change in odour character in olfactometry assessments of abatement techniques.

- Significant differences have been found within the limits indicated by CEN, regarding the individual repeatability, the panel size, the minimum number of rounds. This indicates that further experimental research is necessary to support the revisions of the European standard for dynamic olfactometry and to finetune the application of dynamic olfactometry.

Some recommendations broader than the scope of this thesis are:

- Since only a few reportings exist on the comparison of the different presentation methods of olfactometry (yesno vs dual forced choice), it seems that the comparison of results between the two presentation methods could be further explored.

- More general an update of the odour emission factors seems opportune, taking into account the recent developments in livestock farming and housing systems (24 % of the pigs are now held in ammonia emission low housing systems and the pig farms have increased in size). To assess these emission numbers, the further development of harmonized measurement protocols is required, enabling to reduce also the interlaboratory differences.

- Further exploring the correlations between the different odour measurement techniques to clarify the dose-effect relationship of odour emissions – odour perception and nuisance.

APPENDIX A: SUPPLEMENTARY MATERIAL

Illustration of the retrospectieve screening procedure following CEN (2003)

Odour measurement result of 1 sample, measured with 6 panellists in 3 rounds:

Panellists	ITE Round 1	ΔZ	ITE Round 2	ΔZ	ITE Round 3	ΔZ
1	2896	2.4	362	-3.3	2896	2.4
2	362	-3.3	1448	1.2	362	-3.3
3	724	-1.6	362	-3.3	724	-1.6
4	11585	9.7	5793	4.9	5793	4.9
5	1448	1.2	724	-1.6	1448	1.2
6	362	-3.3	1448	1.2	724	-1.6

Geometric mean of all ITE 's: 1194
Start panel size: 6 panellists

Requirement of CEN (2003) ($-5 \leq \Delta Z \leq 5$): not completely fulfilled, because panellist 4 has an exceeding ITE. Thus all ITE 's of panellist 4 have to be excluded from the calculation of the measurement result, according to the retrospective screening procedure of CEN (2003).

Then the geometric mean and ΔZ values are recalculated on the remaining ITE's. This gives:

Panellists	ITE Round 1	ΔZ	ITE Round 2	ΔZ	ITE Round 3	ΔZ
1	2896	3.5	362	-2.3	2896	3.5
2	362	-2.3	1448	1.7	362	-2.3
3	724	-1.1	362	-2.3	724	-1.1
~~4~~	~~11585~~	~~13.9~~	~~5793~~	~~7.0~~	~~5793~~	~~7.0~~
5	1448	1.7	724	-1.1	1448	1.7
6	362	-2.3	1448	1.7	724	-1.1

Geometric mean of the remaining ITE 's: 832
Requirement CEN (2003): $-5 \leq \Delta Z \leq 5$ fulfilled for remaining ITE

⇨ **Odour concentration: 832 $OU_E\ m^{-3}$**
Final panel size: 5 panellists

APPENDIX B: SUPPLEMENTARY MATERIAL CHAPTER 5

Statistical analysis of variance components of the primary datasets

The variance components on the different levels of the primary data were determined using SAS 9.3 (Proc. Mixed, SAS Institute Inc., NC, USA). Two-level (panellist, round) null-models (intercept only) were fit for individual n-butanol thresholds (dependent variable) of both PPL. The variance components attributed to the sources of variation (panellist and round) in the primary data (individual n-butanol thresholds) of both PPL (datasets 1 & 2) can be found in Table B.1.

Null-models (intercept only models):

$Ygood_{vw} = \beta_0 + \mu_w + e_{vw}$

$Ybest_{vw} = \beta_0 + \mu_w + e_{vw}$

β_0 = intercept

v = round, w = panellist

μ_w = random fault on panellist level

e_{vw}= residual

Intraclass correlation at different levels (percentage variation at the different levels) ρ:

At panellist level: $\rho_i = \sigma^2_{\mu w} / (\sigma^2_{\mu w} + \sigma^2_{evw})*100$

At round level: $\rho_i = \sigma^2_{evw} / (\sigma^2_{\mu w} + \sigma^2_{evw})*100$

$\sigma^2_{\mu w}$ = Variance of the panellist level errors μ_w

σ^2_{evw} = Variance of the round level errors e_{vw}

Table B.1 Variance analysis on the primary datasets of both PPL

Odour	PPL [a]	Source of variation	Variance components from the null-model	
			Var. Est. [b]	% [c]
n-butanol	Good	Panellist	0.009	9
		Round	0.095	91
		Total Variance	0.104	
	Best	Panellist	0.021	31
		Round	0.046	69
		Total Variance	0.067	

[a] Panellists' performance level

[b] Variance estimate

[c] Proportion of the total variance (intraclass correlation)

From the variance components (Table B.1), it can be deduced that the largest proportion of variation is present at the level of rounds for both the good and the best panellists (91 % resp. 69 %). The variation between repetitions of the same panellist (between rounds) is thus larger than the variation between different panellists and this within both PPL. The best panellists also show less variation between rounds (69 %) than the good panellists (91 %), as could be expected, because they have a higher individual repeatability.

Three-level (day, panellist, round) null-models (intercept only) were fit for individual n-butanol thresholds and for individual pig house odour thresholds as dependent variables.

Null-models (intercept only models):

$Ybut_{fgh} = \beta_0 + \mu_h + \mu_{gh} + e_{fgh}$

$Ypig_{fgh} = \beta_0 + \mu_h + \mu_{gh} + e_{fgh}$

β_0 = intercept

f = round, g = panellist, h = sample (day)

μ = random fault on the different levels

e_{fgh}= residual

Intraclass correlation at different levels (percentage variation at the different levels) ρ:

At sample level: $\rho_i = \sigma^2_{\mu h} / (\sigma^2_{\mu h} + \sigma^2_{\mu gh} + \sigma^2_{efgh})*100$

At panellist level: $\rho_i = \sigma^2_{\mu gh} / (\sigma^2_{\mu h} + \sigma^2_{\mu gh} + \sigma^2_{efgh})*100$

At round level: $\rho_i = \sigma^2_{efgh} / (\sigma^2_{\mu h} + \sigma^2_{\mu gh} + \sigma^2_{efgh})*100$

$\sigma^2_{\mu h}$ = Variance of the sample level errors μ_h

$\sigma^2_{\mu gh}$ = Variance of the panellist level errors μ_{gh}

σ^2_{efgh} = Variance of the round level errors e_{fgh}

From the variance components (Table B.2), attributed to the sources of variation (day (sample), panellist and round) in the primary data (individual n-butanol and pig odour thresholds), can be deduced that the largest proportion of variation is present at the level of rounds (i.e. between repetitions of the same panellist) for both n-butanol and pig odour measurements (72 % resp. 76 %). The second largest contribution to the total variation in pig odour thresholds was found at the day level (i.e. between samples of different days), whereas for n-butanol this was situated at the panellist level (i.e. between different panellists). The smallest part of the total variation was at the panellist level for pig odour

and at the day level for n-butanol. It could be expected that the variation due to the day was more important for pig odour than for n-butanol, since the pig odour samples were taken on 4 different days (so under different sampling conditions and thereby presumably having a different odour concentration), while all n-butanol samples analytically had the same concentration (sampled directly from the certified gas bottle in the lab).

Table B.2 Variance analysis on the primary datasets of both odour types

Odour	Source of variation	Variance components from the null-model	
		Var. Est. [a]	**%** [b]
n-butanol	Day (sample)	0.016	8
	Panellist	0.037	19
	Round	0.140	72
	Total Variance	0.194	
pig odour	Day (sample)	0.014	12
	Panellist	0.013	11
	Round	0.085	76
	Total Variance	0.112	

[a] Variance estimate
[b] Proportion of the total variance (intraclass correlation)

APPENDIX C: SUPPLEMENTARY MATERIAL CHAPTER 6

Example of application of annex G of CEN (2003)

Situation:

Technique with expected 40 % efficiency and odour concentration in pig house = 10000 ou_E/m^3

3 samples are taken in the pig house and 3 at the outlet of the aircleaning technique.

Measurement of 3 samples from the outlet with 5 panellists and 3 rounds in the laboratory.

Measurement with 5 panellists and 3 rounds: experimental s_r =0.09 (following chapter 5)

Outlet concentration:

- 10000 ou_E/m^3 – 0.40 . 10000 ou_E/m^3 = 6000 ou_E/m^3
- $\log(conc_{out})$ = 3.778

Calculated confidence interval on outlet concentration:

- Underlimit

$$10^{\left(\log(conc_{out}) - \left(2 \,.\, \frac{s_r}{\sqrt{n}}\right)\right)}$$

$$10^{\left(3.778 - \left(2 \,.\, \frac{0.09}{\sqrt{3}}\right)\right)} = 4710$$

- Upperlimit

$$10^{\left(\log(conc_{out}) + \left(2 \,.\, \frac{s_r}{\sqrt{n}}\right)\right)}$$

$$10^{\left(3.778 + \left(2 \,.\, \frac{0.09}{\sqrt{3}}\right)\right)} = 7643$$

Table C.1 (corresponding with Figure 6.3): Confidence interval on reduction-efficiencies under different measurement-conditions. Confidence interval is determined independent of the concentration at the inlet of the reduction-technique (following Annex H, CEN).

Emission - reduction (%)	n	panel size	rounds	Confidence interval	Width Confidence-interval (%)
90	2	**4**	**2**	83,6 % ≤ 90,0 % ≤ 93,9 %	10
			3	84,3 % ≤ 90,0 % ≤ 93,6 %	9
		5	2	84,2 % ≤ 90,0 % ≤ 93,7 %	10
			3	84,8 % ≤ 90,0 % ≤ 93,4 %	9
		6	2	84,9 % ≤ 90,0 % ≤ 93,4 %	8
			3	85,5 % ≤ 90,0 % ≤ 93,1 %	8
		7	2	85,4 % ≤ 90,0 % ≤ 93,1 %	8
			3	86,0 % ≤ 90,0 % ≤ 92,9 %	7
		8	2	86,1 % ≤ 90,0 % ≤ 92,8 %	7
			3	86,6 % ≤ 90,0 % ≤ 92,6 %	6
	3	**4**	**2**	85,0 % ≤ 90,0 % ≤ 93,3 %	8
			3	85,6 % ≤ 90,0 % ≤ 93,1 %	8
		5	2	85,5 % ≤ 90,0 % ≤ 93,1 %	8
			3	85,9 % ≤ 90,0 % ≤ 92,9 %	7
		6	2	86,0 % ≤ 90,0 % ≤ 92,8 %	7
			3	86,5 % ≤ 90,0 % ≤ 92,6 %	6
		7	2	86,4 % ≤ 90,0 % ≤ 92,6 %	6
			3	86,8 % ≤ 90,0 % ≤ 92,4 %	6
		8	2	86,9 % ≤ 90,0 % ≤ 92,4 %	5
			3	87,3 % ≤ 90,0 % ≤ 92,1 %	5
80	2	**4**	**2**	67,2 % ≤ 80,0 % ≤ 87,8 %	21
			3	68,6 % ≤ 80,0 % ≤ 87,3 %	19
		5	2	68,4 % ≤ 80,0 % ≤ 87,4 %	19
			3	69,6 % ≤ 80,0 % ≤ 86,8 %	17
		6	2	69,9 % ≤ 80,0 % ≤ 86,7 %	17
			3	71,1 % ≤ 80,0 % ≤ 86,2 %	15
		7	2	70,9 % ≤ 80,0 % ≤ 86,3 %	15
			3	72,0 % ≤ 80,0 % ≤ 85,7 %	14
		8	2	72,1 % ≤ 80,0 % ≤ 85,6 %	13
			3	73,1 % ≤ 80,0 % ≤ 85,1 %	12
	3	**4**	**2**	70,1 % ≤ 80,0 % ≤ 86,6 %	17
			3	71,1 % ≤ 80,0 % ≤ 86,2 %	15
		5	2	70,9 % ≤ 80,0 % ≤ 86,3 %	15
			3	71,8 % ≤ 80,0 % ≤ 85,8 %	14
		6	2	72,0 % ≤ 80,0 % ≤ 85,7 %	14
			3	73,0 % ≤ 80,0 % ≤ 85,2 %	12
		7	2	72,8 % ≤ 80,0 % ≤ 85,3 %	12
			3	73,7 % ≤ 80,0 % ≤ 84,8 %	11
		8	2	73,8 % ≤ 80,0 % ≤ 84,7 %	11
			3	74,5 % ≤ 80,0 % ≤ 84,3 %	10

70	2	**4**	**2**	50,8 % ≤ 70,0 % ≤ 81,7 %	31
			3	52,9 % ≤ 70,0 % ≤ 80,9 %	28
		5	2	52,5 % ≤ 70,0 % ≤ 81,0 %	29
			3	54,4 % ≤ 70,0 % ≤ 80,3 %	26
		6	2	54,8 % ≤ 70,0 % ≤ 80,1 %	25
			3	56,6 % ≤ 70,0 % ≤ 79,2 %	23
		7	2	56,3 % ≤ 70,0 % ≤ 79,4 %	23
			3	58,0 % ≤ 70,0 % ≤ 78,6 %	21
		8	2	58,2 % ≤ 70,0 % ≤ 78,5 %	20
			3	59,7 % ≤ 70,0 % ≤ 77,7 %	18
	3	**4**	**2**	55,1 % ≤ 70,0 % ≤ 80,0 %	25
			3	56,7 % ≤ 70,0 % ≤ 79,2 %	23
		5	2	56,4 % ≤ 70,0 % ≤ 79,4 %	23
			3	57,8 % ≤ 70,0 % ≤ 78,7 %	21
		6	2	58,1 % ≤ 70,0 % ≤ 78,5 %	20
			3	59,5 % ≤ 70,0 % ≤ 77,8 %	18
		7	2	59,2 % ≤ 70,0 % ≤ 77,9 %	19
			3	60,5 % ≤ 70,0 % ≤ 77,2 %	17
		8	2	60,7 % ≤ 70,0 % ≤ 77,1 %	16
			3	61,8 % ≤ 70,0 % ≤ 76,4 %	15
60	2	**4**	**2**	34,4 % ≤ 60,0 % ≤ 75,6 %	41
			3	37,2 % ≤ 60,0 % ≤ 74,5 %	37
		5	2	36,7 % ≤ 60,0 % ≤ 74,7 %	38
			3	39,2 % ≤ 60,0 % ≤ 73,7 %	35
		6	2	39,7 % ≤ 60,0 % ≤ 73,5 %	34
			3	42,2 % ≤ 60,0 % ≤ 72,3 %	30
		7	2	41,7 % ≤ 60,0 % ≤ 72,5 %	31
			3	44,0 % ≤ 60,0 % ≤ 71,4 %	27
		8	2	44,3 % ≤ 60,0 % ≤ 71,3 %	27
			3	46,2 % ≤ 60,0 % ≤ 70,2 %	24
	3	**4**	**2**	40,1 % ≤ 60,0 % ≤ 73,3 %	33
			3	42,2 % ≤ 60,0 % ≤ 72,3 %	30
		5	2	41,8 % ≤ 60,0 % ≤ 72,5 %	31
			3	43,7 % ≤ 60,0 % ≤ 71,6 %	28
		6	2	44,1 % ≤ 60,0 % ≤ 71,4 %	27
			3	46,0 % ≤ 60,0 % ≤ 70,4 %	24
		7	2	45,6 % ≤ 60,0 % ≤ 70,6 %	25
			3	47,3 % ≤ 60,0 % ≤ 69,6 %	22
		8	2	47,6 % ≤ 60,0 % ≤ 69,5 %	22
			3	49,1 % ≤ 60,0 % ≤ 68,6 %	20

50	2	**4**	2	18,0 % ≤ 50,0 % ≤ 69,5 %	51
			3	21,5 % ≤ 50,0 % ≤ 68,1 %	47
		5	2	20,9 % ≤ 50,0 % ≤ 68,4 %	48
			3	24,0 % ≤ 50,0 % ≤ 67,1 %	43
		6	2	24,6 % ≤ 50,0 % ≤ 66,8 %	42
			3	27,7 % ≤ 50,0 % ≤ 65,4 %	38
		7	2	27,2 % ≤ 50,0 % ≤ 65,7 %	39
			3	30,0 % ≤ 50,0 % ≤ 64,3 %	34
		8	2	30,4 % ≤ 50,0 % ≤ 64,1 %	34
			3	32,8 % ≤ 50,0 % ≤ 62,8 %	30
	3	**4**	**2**	25,1 % ≤ 50,0 % ≤ 66,6 %	41
			3	27,8 % ≤ 50,0 % ≤ 65,4 %	38
		5	2	27,3 % ≤ 50,0 % ≤ 65,6 %	38
			3	29,6 % ≤ 50,0 % ≤ 64,5 %	35
		6	2	30,1 % ≤ 50,0 % ≤ 64,2 %	34
			3	32,5 % ≤ 50,0 % ≤ 63,0 %	31
		7	2	32,0 % ≤ 50,0 % ≤ 63,2 %	31
			3	34,2 % ≤ 50,0 % ≤ 62,0 %	28
		8	2	34,5 % ≤ 50,0 % ≤ 61,8 %	27
			3	36,3 % ≤ 50,0 % ≤ 60,7 %	24
40	2	4	2	1,6 % ≤ 40,0 % ≤ 63,4 %	62
			3	5,8 % ≤ 40,0 % ≤ 61,8 %	56
		5	2	5,1 % ≤ 40,0 % ≤ 62,1 %	57
			3	8,8 % ≤ 40,0 % ≤ 60,5 %	52
		6	2	9,6 % ≤ 40,0 % ≤ 60,2 %	51
			3	13,3 % ≤ 40,0 % ≤ 58,5 %	45
		7	2	12,6 % ≤ 40,0 % ≤ 58,8 %	46
			3	16,0 % ≤ 40,0 % ≤ 57,2 %	41
		8	2	16,4 % ≤ 40,0 % ≤ 56,9 %	40
			3	19,3 % ≤ 40,0 % ≤ 55,4 %	36
	3	4	2	10,2 % ≤ 40,0 % ≤ 59,9 %	50
			3	13,3 % ≤ 40,0 % ≤ 58,5 %	45
		5	2	12,7 % ≤ 40,0 % ≤ 58,8 %	46
			3	15,5 % ≤ 40,0 % ≤ 57,4 %	42
		6	2	16,1 % ≤ 40,0 % ≤ 57,1 %	41
			3	19,0 % ≤ 40,0 % ≤ 55,6 %	37
		7	2	18,4 % ≤ 40,0 % ≤ 55,9 %	37
			3	21,0 % ≤ 40,0 % ≤ 54,4 %	33
		8	2	21,4 % ≤ 40,0 % ≤ 54,2 %	33
			3	23,6 % ≤ 40,0 % ≤ 52,9 %	29

30	2	4	2	-14,8 % ≤ 30,0 % ≤ 57,3 %	72
			3	-9,8 % ≤ 30,0 % ≤ 55,4 %	65
		5	2	-10,8 % ≤ 30,0 % ≤ 55,8 %	67
			3	-6,4 % ≤ 30,0 % ≤ 54,0 %	60
		6	2	-5,5 % ≤ 30,0 % ≤ 53,6 %	59
			3	-1,2 % ≤ 30,0 % ≤ 51,6 %	53
		7	2	-2,0 % ≤ 30,0 % ≤ 51,9 %	54
			3	1,9 % ≤ 30,0 % ≤ 50,0 %	48
		8	2	2,5 % ≤ 30,0 % ≤ 49,7 %	47
			3	5,9 % ≤ 30,0 % ≤ 47,9 %	42
	3	4	2	-4,8 % ≤ 30,0 % ≤ 53,3 %	58
			3	-1,1 % ≤ 30,0 % ≤ 51,5 %	53
		5	2	-1,8 % ≤ 30,0 % ≤ 51,9 %	54
			3	1,4 % ≤ 30,0 % ≤ 50,3 %	49
		6	2	2,1 % ≤ 30,0 % ≤ 49,9 %	48
			3	5,5 % ≤ 30,0 % ≤ 48,2 %	43
		7	2	4,8 % ≤ 30,0 % ≤ 48,5 %	44
			3	7,8 % ≤ 30,0 % ≤ 46,8 %	39
		8	2	8,3 % ≤ 30,0 % ≤ 46,6 %	38
			3	10,9 % ≤ 30,0 % ≤ 45,0 %	34
20	2	4	2	-31,2 % ≤ 20,0 % ≤ 51,2 %	82
			3	-25,5 % ≤ 20,0 % ≤ 49,0 %	75
		5	2	-26,6 % ≤ 20,0 % ≤ 49,4 %	76
			3	-21,7 % ≤ 20,0 % ≤ 47,4 %	69
		6	2	-20,6 % ≤ 20,0 % ≤ 46,9 %	68
			3	-15,6 % ≤ 20,0 % ≤ 44,6 %	60
		7	2	-16,5 % ≤ 20,0 % ≤ 45,1 %	62
			3	-12,1 % ≤ 20,0 % ≤ 42,9 %	55
		8	2	-11,4 % ≤ 20,0 % ≤ 42,6 %	54
			3	-7,5 % ≤ 20,0 % ≤ 40,5 %	48
	3	4	2	-19,8 % ≤ 20,0 % ≤ 46,6 %	66
			3	-15,6 % ≤ 20,0 % ≤ 44,6 %	60
		5	2	-16,4 % ≤ 20,0 % ≤ 45,0 %	61
			3	-12,6 % ≤ 20,0 % ≤ 43,2 %	56
		6	2	-11,8 % ≤ 20,0 % ≤ 42,8 %	55
			3	-8,1 % ≤ 20,0 % ≤ 40,8 %	49
		7	2	-8,8 % ≤ 20,0 % ≤ 41,2 %	50
			3	-5,3 % ≤ 20,0 % ≤ 39,2 %	45
		8	2	-4,8 % ≤ 20,0 % ≤ 39,0 %	44
			3	-1,9 % ≤ 20,0 % ≤ 37,2 %	39

10	2	4	2	-47,6 % ≤ 10,0 % ≤ 45,1 %	93
			3	-41,2 % ≤ 10,0 % ≤ 42,6 %	84
		5	2	-42,4 % ≤ 10,0 % ≤ 43,1 %	86
			3	-36,9 % ≤ 10,0 % ≤ 40,8 %	78
		6	2	-35,7 % ≤ 10,0 % ≤ 40,3 %	76
			3	-30,1 % ≤ 10,0 % ≤ 37,7 %	68
		7	2	-31,1 % ≤ 10,0 % ≤ 38,2 %	69
			3	-26,1 % ≤ 10,0 % ≤ 35,7 %	62
		8	2	-25,3 % ≤ 10,0 % ≤ 35,4 %	61
			3	-21,0 % ≤ 10,0 % ≤ 33,1 %	54
	3	4	2	-34,8 % ≤ 10,0 % ≤ 39,9 %	75
			3	-30,0 % ≤ 10,0 % ≤ 37,7 %	68
		5	2	-30,9 % ≤ 10,0 % ≤ 38,1 %	69
			3	-26,7 % ≤ 10,0 % ≤ 36,1 %	63
		6	2	-25,8 % ≤ 10,0 % ≤ 35,6 %	61
			3	-21,6 % ≤ 10,0 % ≤ 33,4 %	55
		7	2	-22,4 % ≤ 10,0 % ≤ 33,8 %	56
			3	-18,5 % ≤ 10,0 % ≤ 31,6 %	50
		8	2	-17,9 % ≤ 10,0 % ≤ 31,3 %	49
			3	-14,6 % ≤ 10,0 % ≤ 29,3 %	44

Efficiency (%)	n	N	panel size	d	Width C.I. (%)	ΔWidth C.I. (%)	Personnel meas. cost	ΔCost	ΔCost/ΔWidth	Confidence interval
60	2	4	4	2	41	Ref.	97	Ref.		34,4 % ≤ 60,0 % ≤ 75,6 %
				3	37	-4	145	48		37,2 % ≤ 60,0 % ≤ 74,5 %
			5	2	38	-3	137	40		36,7 % ≤ 60,0 % ≤ 74,7 %
				3	35	-7	206	109		39,2 % ≤ 60,0 % ≤ 73,7 %
			6	2	34	-7	156	59		39,7 % ≤ 60,0 % ≤ 73,5 %
				3	30	-11	234	137		42,2 % ≤ 60,0 % ≤ 72,3 %
			7	2	31	-10	175	78	-8	41,7 % ≤ 60,0 % ≤ 72,5 %
				3	27	-14	262	165		44,0 % ≤ 60,0 % ≤ 71,4 %
			8	2	27	-14	194	97	-7	44,3 % ≤ 60,0 % ≤ 71,3 %
				3	24	-17	291	194		46,2 % ≤ 60,0 % ≤ 70,2 %
	3	6	4	2	33	-8	145	48	-6	40,1 % ≤ 60,0 % ≤ 73,3 %
				3	30	-11	218	121		42,2 % ≤ 60,0 % ≤ 72,3 %
			5	2	31	-11	206	109		41,8 % ≤ 60,0 % ≤ 72,5 %
				3	28	-13	308	211		43,7 % ≤ 60,0 % ≤ 71,6 %
			6	2	27	-14	234	137		44,1 % ≤ 60,0 % ≤ 71,4 %
				3	24	-17	351	254		46,0 % ≤ 60,0 % ≤ 70,4 %
			7	2	25	-16	262	165		45,6 % ≤ 60,0 % ≤ 70,6 %
				3	22	-19	393	297		47,3 % ≤ 60,0 % ≤ 69,6 %
			8	2	22	-19	291	194		47,6 % ≤ 60,0 % ≤ 69,5 %
				3	20	-22	436	339		49,1 % ≤ 60,0 % ≤ 68,6 %
50	2	4	4	2	51		97			18,0 % ≤ 50,0 % ≤ 69,5 %
				3	47	Ref.	145	Ref.		21,5 % ≤ 50,0 % ≤ 68,1 %
			5	2	48	1	137	-8		20,9 % ≤ 50,0 % ≤ 68,4 %
				3	43	-3	206	60		24,0 % ≤ 50,0 % ≤ 67,1 %
			6	2	42	-4	156	11		24,6 % ≤ 50,0 % ≤ 66,8 %
				3	38	-9	234	89		27,7 % ≤ 50,0 % ≤ 65,4 %
			7	2	39	-8	175	30	-4	27,2 % ≤ 50,0 % ≤ 65,7 %
				3	34	-12	262	117		30,0 % ≤ 50,0 % ≤ 64,3 %
			8	2	34	-13	194	48	-4	30,4 % ≤ 50,0 % ≤ 64,1 %
				3	30	-17	291	145		32,8 % ≤ 50,0 % ≤ 62,8 %
	3	6	4	2	41	-5	145	0	0	25,1 % ≤ 50,0 % ≤ 66,6 %
				3	38	-9	218	73		27,8 % ≤ 50,0 % ≤ 65,4 %
			5	2	38	-8	206	60		27,3 % ≤ 50,0 % ≤ 65,6 %
				3	35	-12	308	163		29,6 % ≤ 50,0 % ≤ 64,5 %
			6	2	34	-12	234	89		30,1 % ≤ 50,0 % ≤ 64,2 %
				3	31	-16	351	206		32,5 % ≤ 50,0 % ≤ 63,0 %
			7	2	31	-15	262	117		32,0 % ≤ 50,0 % ≤ 63,2 %
				3	28	-19	393	248		34,2 % ≤ 50,0 % ≤ 62,0 %
			8	2	27	-19	291	145		34,5 % ≤ 50,0 % ≤ 61,8 %
				3	24	-22	436	291		36,3 % ≤ 50,0 % ≤ 60,7 %

LIST OF REFERENCES

Andersen, K. B. (2013). Chemical assessment of non-thermal plasma for reduction of odour emissions from pig houses. Technical report BCE -TR-5, Department of Engineering, Aarhus University. Denmark. 97 pp.

Andersen, K. B., Glasius, M., & Feilberg, A. (2014). Gas–particle partitioning of odorants in a pig house measured by thermal desorption GC/MS. *Environmental Science: Processes & Impacts*, 16(5), 1059-1068.

Aneja, V. P., Schlesinger, W. H., & Erisman, J. W. (2009). Effects of Agriculture upon the Air Quality and Climate: Research, Policy, and Regulations. *Environmental Science & Technology*, 43, 4234-4240.

Bereznicki, S. D., Heber, A. J., Akdeniz, N., Jacobson, L. D., Hetchler, B. P., Heathcote, K. Y. et al. (2012). Odor and Odorous Chemical Emissions from Animal Buildings: Part 1. Project Overview, Collection Methods, and Quality Control. *Transactions of the Asabe*, 55, 2325-2334.

Bilsen I., De Fré R., Bosmans S (2008) Code van goede praktijk bepalen van de geurverspreiding door middel van snuffelploegmetingen, 2008/MIM/R/022.

Blanes-Vidal, V., Hansen, M. N., Adamsen, A. P. S., Feilberg, A., Petersen, S. O., & Jensen, B. B. (2009a). Characterization of odor released during handling of swine slurry: Part I. Relationship between odorants and perceived odor concentrations. *Atmospheric Environment*, 43, 2997-3005.

Blanes-Vidal, V., Hansen, M. N., Adamsen, A. P. S., Feilberg, A., Petersen, S. O., & Jensen, B. B. (2009b). Characterization of odor released during handling of swine slurry: Part II. Effect of production type, storage and physicochemical characteristics of the slurry. *Atmospheric Environment*, 43, 3006-3014.

Blazy, V., de Guardia, A., Benoist, J.C., Daumoin, M. , Guiziou, F. , Lemasle, M., Wolbert, D., Barrington, S., (2015) Correlation of chemical composition and odor concentration for emissions from pig slaughterhouse sludge composting and storage, *Chemical Engineering Journal* 276: 398–409.

Boeker, P. (2014). On 'Electronic Nose' methodology. *Sensors and Actuators* B, 204, 2-17.

Boeker, P. & Haas, T. (2007). Die Messunsicherheit der Olfaktometrie. *Gefahrstoffe Reinhaltung der Luft*, 67, 331-339.

Boeker, P., Haas, T., Diekmann, B., & Schulze Lammers, P. (2008). A Monte-Carlo simulation of the measurement uncertainty of olfactometry. *Chemical Engineering Transactions*, 15, 109-114

Bokowa, A., 2010. The effect of sampling on the measured odour concentration. *Chemical Engineering Transactions*, 23, 43-48.

Bokowa,A. 2012. Odour assessment: determining the optimum temperature and time for Tedlar sampling bag pre-conditioning. *Water Science and Technology* 66:1806-1811.

Brattoli, M., Barbieri, G., Barbieri, P., Cozzutto, S., de Gennaro, G., Fabbris, A. et al. (2014). Development and technology assessment of the analytical performance of an eight position dynamic olfactometer. *Chemical Engineering Transactions*, 40, 115-120.

Brattoli, M., de Gennaro, G., de Pinto, V., Loiotile, A. D., Lovascio, S., & Penza, M. (2011). Odour Detection Methods: Olfactometry and Chemical Sensors. *Sensors*, 11, 5290-5322.

Bruneel, J., Walgraeve, C., Van Huffel, K., & Van Langenhove, H. (2016). Determination of the gas-to-liquid partitioning coefficients using a new dynamic absorption method (DynAb method). *Chemical Engineering Journal*, 283, 544-552.

Buck, L. (2004) Olfactory Receptors and Odor Coding in Mammals; *Nutrition Reviews*; Nov 2004; 62, 11 (II)S184-S188.

Bullers, S. (2005). Environmental stressors, perceived control, and health: The case of residents near large-scale hog farms in eastern North Carolina. *Human Ecology*, 33, 1-16.

Bulliner, Koziel, Cai, Wright, (2006) Characterization of Livestock Odors Using Steel Plates, Solid-Phase Microextraction, and Multidimensional Gas Chromatography–Mass Spectrometry–Olfactometry. *J. Air & Waste Manage. Assoc.* 56:1391–1403.

Cai ,L.; Koziel, J. A.; Loa, Y-C; Hoff, S.J., Characterization of volatile organic compounds and odorants associated with swine barn particulate matter using solid-phase microextraction and gas chromatography–mass spectrometry–olfactometry. *Journal of Chromatography A*, 1102 (2006) 60–72.

Capelli, L., Sironi, S., Del Rosso, R., Centola, P., & Il Grande, M. (2008). A comparative and critical evaluation of odour assessment methods on a landfill site. *Atmospheric Environment*, 42, 7050-7058.

Capelli, L., Sironi, S., Del Rosso, R., Centola, P., & Bonati, S. (2010). Improvement of olfactometric measurement accuracy and repeatability by optimization of panel selection procedures. *Water Science and Technology*, 61, 1267-1278.

Capelli, L., Sironi, S., Del Rosso, R., Centola, P., Rossi, A., & Austeri, C. (2011). Olfactometric approach for the evaluation of citizens' exposure to industrial emissions in the city of Terni, Italy. *Science of the Total Environment*, 409, 595-603.

Capelli, L., Sironi, S., Del Rosso, R., Bianchi, G., & Davoli, E. (2012). Olfactory and toxic impact of industrial odour emissions. *Water Science and Technology*, 66, 1399-1406.

CEN (2003). Air Quality. Determination of odour concentration by dynamic olfactometry (European Standard EN 13725:2003, 70p). *European Committee for standardization*.

Clanton, C. J., Schmidt, D. R., Nicolai, R. E., Goodrich, P. R., Jacobson, L. D., Janni, K. A. et al. (1999). Dynamic olfactometry variability in determining odor dilutions-to-threshold. *Transactions of the Asae*, 42, 1103-1112.

Cole, D., Todd, L., & Wing, S. (2000). Concentrated swine feeding operations and public health: A review of occupational and community health effects. *Environmental Health Perspectives*, 108, 685-699.

De Bruyn, G., Baron, M., Van Langenhove, H., Hendriks, J., Andries, A., Saevels, P. et al. (2001). Development of a straightforward procedure to determine odour and ammonia emissions from agricultural constructions for adapted environmental regulations in Flanders Research Project, Belgium (in Dutch).

Defoer, N. & Van Langenhove, H. (2004). Variability and repeatability of olfactometric results of n-butanol, pig odour and a synthetic gas mixture. *Water Science and Technology*, 50, 65-73.

Dohoo, I., Martin, W., & Stryhn, H. (2010). *Veterinary epidemiologic research*. AVC Inc., Charlottetown, Prince Edward Island.

Donham, K. J. (2010). Community and occupational health concerns in pork production: A review. *Journal of Animal Science*, 88, E102-E111.

Donham, K. J., Wing, S., Osterberg, D., Flora, J. L., Hodne, C., Thu, K. M. et al. (2007). Community health and socioeconomic issues surrounding concentrated feeding operations. *Environmental Health Perspectives*, 115, 317-320.

Driesen, K., Van Elst, T., Demeyer, P., & Brusselman, E. (2016). Geuremissies en hinder in de Nederlandse varkenshouderij, onderzoek in opdracht van de Nederlandse Vakbond Varkenshouders (Rep. No. RapportILVO1688_15_0083).

Environment Agency (1995). Determination of Odour Index and Odour Emission Rate. *Environment Agency, Tokyo, Japan, Notification No.63*.

Eyckmans, J., De Jaeger, S., Rousseau, S. (2011) Hedonic valuation of odor nuisance using field measurements, a case study of an animal waste processing facility in Flanders, *HUB research papers 2011/19, Economics & Management*, 26p.

EN 16841-1:2016 Ambient air - Determination of odour in ambient air by using field inspection - Part 1: Grid method

EN 16841-2:2016 Ambient air - Determination of odour in ambient air by using field inspection - Part 2: Plume method

European Commission (2003) Integrated Pollution Prevention and Control (IPPC). Reference document on Best Available Techniques for Intensive Rearing of Poultry and Pigs July 2003.

Feilberg,A., D.Liu, A.P.S.Adamsen, M.J.Hansen, and K.E.N.Jonassen. 2010. Odorant Emissions from Intensive Pig Production Measured by Online Proton-Transfer-Reaction Mass Spectrometry. *Environ. Sci. Technol.* 47:5894-5900.

Friedrich, M. & Kosmider, J. (2012). Precision of Odour Abatement Efficiency Determination in Changing Conditions. *Chemical Engineering Transactions*, 30, 265-270.

Gostelow, P., Parsons, S. A., & Stuetz, R. M. (2001). Odour measurements for sewage treatment works. *Water Research*, 35, 579-597.

Guillot,J.M., andS.Beghi. 2008. Permeability to water and hydrogen sulphide of some sampling bags recommended by EN13725. *Chemical Engineering Transactions* 15:79-85.

Gutierrez, M. C., Chica, A. F., Martin, M. A., & Romain, A. C. (2014). Compost Pile Monitoring Using Different Approaches: GC-MS, E-nose and Dynamic Olfactometry. *Waste and Biomass Valorization*, 5, 469-479.

Gutierrez, M. C., Martin, M. A., Pagans, E., Vera, L., Garcia-Olmo, J., & Chica, A. F. (2015). Dynamic olfactometry and GC-TOFMS to monitor the efficiency of an industrial biofilter. *Science of the Total Environment*, 512, 572-581.

Hangartner, M. (1986). Selection and treatment of panelists for determination of odor thresholds.

Hammond, E. G., Fedler, C., & Smith, R. J. (1981). Analysis of particle-borne swine house odors. *Agriculture and Environment*, 6(4), 395-401.

Hansen, M. J., Adamsen, A. P. S., & Feilberg, A. (2013). Recovery of odorants from an olfactometer measured by proton-transfer-reaction mass spectrometry. *Sensors*, 13, 7860-7871.

Hansen,M.J., A.Feilberg, and A.P.S.Adamsen. 2010. Stability of volatile reduced sulphur compounds in the dilution system of an olfactometer. *Chemical Engineering Transactions* 23:67-72.

Hansen, M. J., Adamsen, A. P. S., Feilberg, A., & Jonassen, K. E. N. (2011). Stability of odorants from pig production in sampling bags for olfactometry. *Journal of Environmental Quality*, 40, 1096-1102.

Hansen, M. J., Adamsen, A. P. S., Pedersen, P., & Feilberg, A. (2012). Prediction of Odor from Pig Production Based on Chemical Odorants. *Journal of Environmental Quality*, 41, 436-443.

Hansen, M. J., Jonassen, K. E. N., & Feilberg, A. (2014). Evaluation of abatement technologies for pig houses by dynamic olfactometry and on-site mass spectrometry. *Chemical Engineering Transactions*, 40, 253-258.

Hansen, Jonassen, LØkke, Adamsen and Feilberg, (2016) Multivariate prediction of odor from pig production based on in-situ measurement of odorants, *Atmospheric Environment* 135: 50-58

Hayes, E. T., Curran, T. P., & Dodd, V. A. (2006a). Odour and ammonia emissions from intensive pig units in Ireland. *Bioresource Technology*, 97, 940-948.

Hayes, E. T., Curran, T. P., & Dodd, V. A. (2006b). Odour and ammonia emissions from intensive poultry units in Ireland. *Bioresource Technology*, 97, 933-939.

Hayes, J. E., Stevenson, R. J., & Stuetz, R. M. (2014). The impact of malodour on communities: A review of assessment techniques. *Science of the Total Environment*, 500, 395-407.

Heynderickx, P. M., Van Huffel, K., Dewulf, J., & Van Langenhove, H. (2013). Application of similarity coefficients to SIFT-MS data for livestock emission characterization. *Biosystems Engineering*, 114, 44-54.

Heynderickx, P. M., Van Huffel, K., Dewulf, J., & Van Langenhove, H. (2012) SIFT-MS for livestock emission characterization: application of similarity coefficients *Chemical Engineering Transactions*, 30, 157-162.

Higuchi, T. (2009). Estimation of uncertainty in olfactometry. *Water Science and Technology*, 59, 1409-1413.

Hogberg, M. G., Fales, S. L., Kirschenmann, F. L., Honeyman, M. S., Miranowski, J., & Lasley, P. (2005). Interrelationships of animal agriculture, the environment, and rural communities. *Journal of Animal Science*, 83, E13-E17.

Hove, N. C. Y., Demeyer, P., Van der Heyden C., Van Weyenberg, S., & Van Langenhove, H. (2017). Improving the repeatability of dynamic olfactometry according to EN 13725: A case study for pig odour. *Biosystems Engineering*, 161, 70-79.

Hove, N., Van Langenhove, H., & Demeyer, P. (2012). Development of an olfactometric measuring facility according to CEN EN 13725 and to generate up to date odour concentrations from animal houses in Flanders. *Chemical Engineering Transactions*, 30, 97-102.

Hove, N. C. Y., Van Langenhove, H., Van Weyenberg, S., & Demeyer, P. (2016). Comparative odour measurements according to EN 13725 using pig house odour and n-butanol reference gas. *Biosystems Engineering*, 143, 119-127.

Jacobson L.D., Hetchler, B.P., Schmidt, D.R., Nicolai, R.E., Heber, A.J., Ni, J-Q, Hoff, S.J., Koziel, J.A., Zhang, Y., Beasly, D.B., Parker, D.B., (2008) Quality Assured Measurements of Animal Building Emissions: Odor Concentrations, *J. Air & Waste Manage. Assoc.* 58:806–811.

ISO (1995) Guide to the Expression of Uncertainty in Measurement, International Organization for Standardization, Geneva, Switzerland, ISO/IEC Guide 98:1995

Jiang, John;Coffey, Patrick;Toohey, Brendan (2006), Improvement of Odor Intensity Measurement Using Dynamic Olfactometry. *Journal of the Air & Waste Management Association*; 56, 5; pg. 675-683

Jonassen, K. E. N., Nielsen, M. B. F., & Hansen, M. J. (2014). Online or Delayed Olfactometry – a Comparison Based on Results from Four Different Types of Pig Housing or Mitigation Systems. *Chemical Engineering Transactions*, 40, 7-12.

Jonassen, K. E. N., Pedersen, P., Riis, A. L., & Sorensen, K. (2012). Does the choice of olfactometric laboratory affect the efficiency of odour abatement technologies? *Chemical Engineering Transactions*, 30, 43-48.

Kai, P. & Schäfer, A. (2004). Identification of Key Odour Components in Pig House Air using Hyphenated Gas Chromatography Olfactometry. Agricultural Engineering International: the *CIGR Journal of Scientific Research and Development*, VI. , 1-11, December, 2004.

Keller Andreas, Hempstead Margaret, Gomez Iran A, Gilbert Avery N and Vosshall Leslie B (2012) An olfactory demography of a diverse metropolitan population. BMC *Neuroscience* 2012, 13:122.

Ki-Hyun Kim, Ji-Won Ahn, Ye-Jin Choi, Hang T. Nguyen, (2006) The loss patterns of reduced sulfur compounds in contact with different tubing materials Journal of *Chromatography A*, 1132 (2006) 228–233

Ki-Hyun Kim, Gyoo-Hoon Choi , Ye-Jin Choi , Hee-Nam Songb, H.-S. Yang , J.-M. Oh (2006) The effects of sampling materials selection in the collection of reduced sulfur compounds in air *Talanta* 68: 1713–1719

Kim, K. H. & Park, S. Y. (2008). A comparative analysis of malodor samples between direct (olfactometry) and indirect (instrumental) methods. *Atmospheric Environment*, 42, 5061-5070.

Koziel,J.A., J.P.Spinhirne, J.D.Lloyd, D.B.Parker, D.W.Wright, and F.W.Kuhrt. 2005. Evaluation of sample recovery of malodorous livestock gases from air sampling bags, solid-phase microextraction fibers, Tenax TA sorbent tubes, and sampling canisters. *Journal of the Air & Waste Management Association* 55:1147-1157.

Klarenbeek, J. V., Ogink, N. W. M., & van der Voet, H. (2014). Odor measurements according to EN 13725: A statistical analysis of variance components. *Atmospheric Environment*, 86, 9-15.

Koziel,J.A., J.P.Spinhirne, J.D.Lloyd, D.B.Parker, D.W.Wright, and F.W.Kuhrt. 2005. Evaluation of sample recovery of malodorous livestock gases from air sampling bags, solid-phase microextraction fibers, Tenax TA sorbent tubes, and sampling canisters. *Journal of the Air & Waste Management Association* 55:1147-1157.

Laor, Y., Ozer, Y., Hanan, A., Orenstein, P., (2010). Methodological aspects of sample collection for dynamic olfactometry. *Chemical Engineering Transactions* 23: 55-50.

Laor, Y., Orenstein, P., Ravid, U., Baybikov, R., Hanan, A., Saadi, I., Abbou, Y. (2011). A Screening Tool for Selection of Field Odor Assessors. *Journal of the Air & Waste Management Association*, 61:12, 1353-1360, DOI: 10.1080/10473289.2011.595989.

Laor, Y., Parker, D., & Pagé, T. (2014). Critical elements in the measurement, monitoring and prediction of odors in the environment. *Chemical Engineering Transactions*, 40, 247-252.

Laor, Y., Parker, D., & Page, T. (2014). Measurement, prediction, and monitoring of odors in the environment: a critical review. *Reviews in Chemical Engineering*, 30(2), 139-166.

Laska, M. & Hudson, R. (1991). A Comparison of the Detection Thresholds of Odor Mixtures and Their Components. *Chemical Senses*, 16(6), 651-662.

P. D. Le, A. J. A. Aarnink, N. W. M. Ogink, M. W. A. Verstegen (2005) EFFECTS OF ENVIRONMENTAL FACTORS ON ODOR EMISSION FROM PIG MANURE. *Transactions of the ASAE*. Vol. 48(2): 757−765.

Leffingwell J.C., Olfaction –V, *Leffingwell Reports*, Vol. 2, (No. 1), mei 2002

Leonardos, G. (1995). Review of odor control regulations in the USA. In Odors, Indoor and Environmental Air, *Proceedings of a Specialty Conference of the Air and Waste Management Association*, Bloomington, MN, 73-84.

Libby, L. W. & Sharp, J. S. (2003). Land-use compatibility, change, and policy at the rural-urban fringe: Insights from social capital. *American Journal of Agricultural Economics*, 85, 1194-1200.

LNE (2011). Geactualiseerd richtlijnenboek milieueffectenrapportage "Basisrichtlijnen per activiteitengroep: Landbouwdieren" (in Dutch). Brussels, Belgium: Departement Leefmilieu, Natuur en Energie (LNE), Afdeling Milieu, Natuur- en Energiebeleid, Dienst MER.

LNE (2010) Behandeling van geurklachten door lokale overheden, 82 p.

LNE (2012) Richtlijnenboek Lucht, 212 p.

LNE (2017) IMPACT handleiding, 79p.

Mackie RI, Stroot PG and Varel VH (1998) Biochemical identification and biological origin of key odor components in livestock waste. *J ANIM SCI*, 76:1331-1342.

Mair, S., Witek, W., Dahn, U., Mayer, F., & Breuer, K. (2006). About precision in human olfactometric measurements. *Gefahrstoffe Reinhaltung der Luft*, 66, 94-98.

Malnic B., Hirono J., Sato T., Buck L.B. (1999) Combinatorial Receptor Codes for Odors. *Cell*, Vol. 96, 713–723, March 5, 1999.

Mannebeck, D. & Mannebeck, H. (2001). Interlaboratory comparison of dynamic olfactometry in Central Europe 2000. *Water Science and Technology*, 44, 27-32.

Mannebeck, D., 2017; Olfactometers according to EN 13725; part C, p545-551 in: Springer Handbook of Odor, Springer Dordrecht Heidelberg London New York, ISBN: 978-3-319-26930-6, 1137pp.

Martens, W., Martinec, M., Zapirain, R., Stark, M., Hartung, E., & Palmgren, U. (2001). Reduction potential of microbial, odour and ammonia emissions from a pig facility by biofilters. *International Journal of Hygiene and Environmental Health*, 203, 335-345.

Maxeiner, B., & Mannebeck, D. (2004). Round robin test olfactometry 2003. *GEFAHRSTOFFE REINHALTUNG DER LUFT*-GERMAN EDITION-, 118-123.

Maxeiner B., 2016, Proficiency test for dynamic olfactometry with real odour, Poster, 6th IWA Conference on Odours & Air Emissions, Paris, DOI:10.13140/RG.2.1.3116.0402

Maxeiner B., (2006) Olfactometric interlaboratory comparison test 2005. *Water Environment Federation*, WEF/A&WA Odors and Air Emissions 2006, pp. 688–699.

McGinley, M. A. & McGinley, C. M. (1999) Odor Evaluation Fundamentals and Applications for Indoor Air Quality Research. *Proceedings of The First NSF International Conference on Indoor Air Health*, Denver, CO: 3-5 May 1999.

McGinley, C. M. (2002). Standardized odor measurement practices for air quality testing. In *Proceedings of the Air and Waste Management Association*. Symposium on Air Quality Measurement Methods and Technology - November 13-15, 2002, San Francisco.

McGinley, C. M., & McGinley, M. A. (2006). An Odor Index Scale for Policy and Decision Making Using Ambient & Source Odor Concentrations. *Proceedings of the Water Environment Federation*, 2006(3), 244-250

McGinley, M. A. & McGinley, C. M. (2006). Precision of olfactometry and odor testing results. *Proceedings of the Water Environment Federation*, 2006, 657-666.

McGinley, M. A. & McGinley, C. M. (2010). Odor assessor performance to reference and non-reference odorants. *Proceedings of the Water Environment Federation*, 2010, 240-251.

Melse, R. W. & Mol, G. (2004). Odour and ammonia removal from pig house exhaust air using a biotrickling filter. *Water Science and Technology*, 50, 275-282.

Melse, R. W., Ogink, N. W. M., & Rulkens, W. H. (2009a). Air Treatment Techniques for Abatement of Emissions from Intensive Livestock Production. *The Open Agriculture Journal*, 3, 6-12.

Melse, R. W., Ogink, N. W. M., & Rulkens, W. H. (2009b). Overview of European and Netherlands' regulations on airborne emissions from intensive livestock production with a focus on the application of air scrubbers. *Biosystems Engineering*, 104, 289-298.

Mielcarek, P. & Rzenik, W. (2015). Odor emission factors from livestock production. Polish *Journal of Environmental Studies*, 24, 27-35.

Miller, G. Y., Maghirang, R. G., Riskowski, G. L., Heber, A. J., Robert, M. J., & Muyot, M. E. T. (2004). Influences on air quality and odor from mechanically ventilated swine finishing buildings in Illinois. *Journal of Food Agriculture & Environment*, 2, 353-360.

Mirabelli, M. C., Wing, S., Marshall, S. W., & Wilcosky, T. C. (2006). Asthma symptoms among adolescents who attend public schools that are located near confined swine feeding operations. *Pediatrics*, 118, E66-E75.

Mochalski,P., B.Wzorek, I.Sliwka, and A.Amann. 2009. Suitability of different polymer bags for storage of volatile sulphur compounds relevant to breath analysis. Journal of *Chromatography B* 877:189-196.

Mol, G. & Ogink, N. W. M. (2002). Geuremissies uit de veehouderij II: Overzichtsrapportage 2000-2002. (Rep. No. 2002-09). Wageningen: IMAG.

Moulton, D. G. (1975). Cell renewal in the olfactory epithelium of the mouse. In *Olfaction and Taste* 5 (pp. 111-114). New York: Academic Press.

Munoz, R., Sivret, E. C., Parcsi, G., Lebrero, R., Wang, X. G., Suffet, I. H. et al. (2010). Monitoring techniques for odour abatement assessment. *Water Research*, 44, 5129-5149.

Nagata,Y. 2003. Measurement of odor threshold by triangle odor bag method. p. 118-127. Odor measurement review. Ministry of the Environment, Goverment of Japan.

Ngwabie,N.M., G.W.Schade, T.G.Custer, S.Linke, and T.Hinz. 2008. Abundances and Flux Estimates of Volatile Organic Compounds from a Dairy Cowshed in Germany. *J Environ Qual* 37:565-573.

Ji-Qin Ni, Wayne P. Robarge, Changhe Xiao , Albert J. Heber, (2012) Volatile organic compounds at swine facilities: A critical review, *Chemosphere* 89, 769–788

Nicell, J. A. (2009). Assessment and regulation of odour impacts. *Atmospheric Environment*, 43, 196-206.

Nicolai et al. 1997 Development of a dynamic olfactometer lab, Paper 974019, *An ASAE Meeting presentation*.

Nicolas, J., Delva, J., Cobut, P., & Romain, A. C. (2008). Development and validating procedure of a formula to calculate a minimum separation distance from piggeries and poultry facilities to sensitive receptors. *Atmospheric Environment*, 42, 7087-7095.

Nimmermark, S. (2004). Odour influence on well-being and health with specific focus on animal production emissions. *Annals of Agricultural and Environmental Medicine*, 11, 163-173.

Nimmermark, S. (2011). Influence of odour concentration and individual odour thresholds on the hedonic tone of odour from animal production. *Biosystems Engineering*, 108, 211-219.

Nimmermark, S. A., Jacobson, L. D., Schmidt, D. R., & Gay, S. W. (2005). Predictions by the Odor From Feedlots, Setback Estimation Tool (OFFSET) compared with observations by neighborhood monitors. *Journal of the Air & Waste Management Association*, 55, 1306-1314.

Ogink, N. W. M. (2010). Assignment of odour emission factors in the Dutch regulation on odour nuisance in animal production based on odour emission research. (Rep. No. 391 (in Dutch)).

Ogink, N. W. M., Keen, A., & Klarenbeek, J. V. (1995). Simulation as a research tool for improving accuracy and precision in olfactometry. In *Proc. Int. Livestock Odor Conference* 1995 (pp. 35-39). Ames, Iowa: Iowa State University of Science and Technology (USA).

Parker, D. B., Rhoades, M. B., Schuster, G. L., Koziel, J. A., & Perschbacher-Buser, Z. L. (2005). Odor characterization at open-lot beef cattle feedyards using triangular forced-choice olfactometry. *Transactions of the Asae*, 48, 1527-1535.

Parker,D.B., Z.L.Perschbacher-Buser, N.A.Cole, and J.A.Koziel. 2010. Recovery of Agricultural Odors and Odorous Compounds from Polyvinyl Fluoride Film Bags. *Sensors* 10:8536-8552.

Pillai, S. M., Parcsi, G., Wang, X., Gallagher, E., Dunlop, M., & Stuetz, R. M. (2010). Assessment of direct headspace analysis of broiler chicken litter odorants. *Chemical Engineering Transactions*, 23, 207-212.

Qu, G., Feddes, J. J. R., Leonard, J. J., Coleman, R. N., & Armstrong, W. W. (2001). Normalization of the olfactory response of an odor panel. *Transactions of the Asae*, 44, 1833-1838.

Qu, G. L. & Feddes, J. J. R. (2007). Development of a reference artificial swine odor. *Transactions of the Asabe*, 50, 1789-1793.

Radon, K., Peters, A., Praml, G., Ehrenstein, V., Schulze, A., Hehl, O. et al. (2004). Livestock odours and quality of life of neighbouring residents. *Annals of Agricultural and Environmental Medicine*, 11, 59-62.

Romain, A. C., Nicolas, J., Cobut, P., Delvat, J., Nicks, B., & Philippe, F. X. (2013). Continuous odour measurement from fattening pig units. *Atmospheric Environment*, 77, 935-942.

Rzenik, W., Mielcarek, P., & Jugowar, J. L. (2014). The Emission of Odor from Livestock Buildings for Dairy Cattle in Poland. *Applied Engineering in Agriculture*, 30, 961-970.

Schamp, N. & Van Langenhove, H. (1987). Geurhinder. [27], 1-247. Kapellen, DNB/Uitgeverij Pelckmans. *Monografieën Leefmilieu Nu*.

Schiffman, S. S. , Bennett, J.L., Raymer, J.H. (2001) Quantification of odors and odorants from swine operations in North Carolina, *Agricultural and Forest Meteorology* 108: 213–240.

Sulyok,M., C.Haberhauer-Troyer, E.Rosenberg, and M.Grasserbauer. 2001. Investigation of the storage stability of selected volatile sulfur compounds in different sampling containers. *Journal of Chromatography* A 917:367-374.

Schinasi, L., Horton, R. A., Guidry, V. T., Wing, S., Marshall, S. W., & Morland, K. B. (2011). Air Pollution, Lung Function, and Physical Symptoms in Communities Near Concentrated Swine Feeding Operations. *Epidemiology*, 22, 208-215.

Sironi, S., Capelli, L., Centola, P., Del Rosso, R., & Il Grande, M. (2006). Odour emission factors for the prediction of odour emissions from plants for the mechanical and biological treatment of MSW. *Atmospheric Environment*, 40, 7632-7643.

Sironi, S., Capelli, L., Centola, P., Del Rosso, R., & Pierucci, S. (2010). Odour impact assessment by means of dynamic olfactometry, dispersion modelling and social participation. *Atmospheric Environment*, 44, 354-360.

Sneath, R. W. (2003). Quality control of olfactometry at SRI and in Europe. In *Odor Measurement Review* (pp. 82-87). Ministry of the Environment, Government of Japan.

Su, C. Y., Menuz, K., & Carlson, J. R. (2009). Olfactory perception: receptors, cells, and circuits. *Cell*, 139(1), 45-59.

Sundberg, C., Yu, D., Franke-Whittle, I., Kauppi, S., Smars, S., Insam, H. et al. (2013). Effects of pH and microbial composition on odour in food waste composting. *Waste Management*, 33, 204-211.

Thu, K. M. (2002). Public health concerns for neighbors of large-scale swine production operations. *Journal of Agricultural Safety and Health*, 8, 175-178.

Trabue, S., Kerr, B., Bearson, B., & Ziemer, C. (2011). Swine Odor Analyzed by Odor Panels and Chemical Techniques. *Journal of Environmental Quality*, 40, 1510-1520.

Trabue,S.L., J.C.Anhalt, and J.A.Zahn. 2006. Bias of Tedlar bags in the measurement of agricultural odorants. *J Environ Qual* 35:1668-1677.

Ubeda, Y., Lopez-Jimenez, P. A., Nicolas, J., & Calvet, S. (2013). Strategies to control odours in livestock facilities: a critical review. *Spanish Journal of Agricultural Research*, 11, 1004-1015.

Ulens, T. (2015) Source-oriented techniques for improving indoor air quality ans assessment of air emissions from pig husbandry. PhD Thesis. Ghent University. Belgium.

Van Boheemen, A. (2012) Comparison of Odour Concentrations Obtained by Yes/No and 2AFC Mode, *Chemical Engineering Transactions*, Vol. 30, ISBN 978-88-95608-21-1; ISSN 1974-9791.

Van Broeck, G. & Van Langenhove, H. (2000). Onderzoek Geurnormering. Ontwikkelen van een methode voor het opstellen van een geurnormering per bedrijf. Evaluatie van de toegepaste methode. Vakgroep Organische Chemie, Faculteit Landbouwkundige en Toegepaste Biologische Wetenschappen, Universiteit Gent.

Van der Heyden, C., Demeyer, P., & Volcke, E. I. P. (2015). Mitigating emissions from pig and poultry housing facilities through air scrubbers and biofilters: State-of-the-art and perspectives. *Biosystems Engineering*, 134, 74-93.

Van Durme Jim & Werbrouck Bas (2015) Phase ratio variation approach for the study of partitioning behavior of volatile organic compounds in polymer sample bags: Nalophan case study *Environ Sci Pollut Res* (2015) 22:11067–11075

Van Elst, T. (2013) Round robin test environmental odours Testing for ageing of samples, powerpoint presentatie 20131106.

Van Elst, T. & Delva, J. (2016). The European standard prEN16841-2 (determination of odour in ambient air by using field inspection: plume method): a review of 20 year experience with the method in Belgium. *Chemical Engineering Transactions*, 54, 175-180.

Van Harreveld, A. P. (2014). Progress in the review of EN 13725: focus on sampling and uncertainty. *Chemical Engineering Transactions*, 40.

Van Harreveld, A. P. & Heeres, P. (1995). Quality-Control and Optimization of Dynamic Olfactometry Using N-Butanol As A Standard Reference Odorant. *Staub Reinhaltung der Luft*, 55, 45-50.

Van Harreveld, A. P. & Heeres, P. (1997). The validation of the draft European CEN standard for dynamic olfactometry by an interlaboratory comparison on n-butanol. *Gefahrstoffe Reinhaltung der Luft*, 57, 393-398.

Van Harreveld, A. P., Mannebeck, D., & Maxeiner, B. (2009). Proficiency testing as the key element in implementing EN13275 olfactometry. *Water Science and Technology*, 59, 1649-1655.

Van Huffel, K., Heynderickx, P. M., Dewulf, J., & Van Langenhove, H. (2012). Measurement of odorants in livestock buildings: SIFT-MS and TD-GC-MS. *Chemical Engineering Transactions*, 30, 67-72.

Van Huffel, K., Hansen, M. J., Feilberg, A., Liu, D., Van Langenhove, H. (2016) Level and distribution of odorous compounds in pig exhaust air from combined room and pit ventilation, *Agriculture, Ecosystems and Environment* 218 : 209–219.

Van Langenhove, H. & De Bruyn, G. (2001). Development of a procedure to determine odour emissions from animal farming for regulatory purposes in Flanders. *Water Science and Technology*, 44, 205-210.

Van Langenhove, H. & Defoer, N. (2002). Validation of the measurement procedure to determine odour and ammonia emissions from reference intensive farming houses to support the implementation of the directive of the assessment procedure for low ammonia emission housing systems VLM/ANIMAL/TWOL2001/mjp2000-21/88, Belgium (in Dutch).

Van Langenhove H., Van Elst T., De Roo K., Philips G. & Bossuyt M. (2007) Milieurapport Vlaanderen, Achtergronddocument 2007, geurhinder, Vlaamse Milieumaatschappij

Van Ransbeeck, N. (2013), Particulate matter, ammonia and greenhouse gases in pig fattening facilities: measuring strategies, indoor concentrations and emissions. PhD thesis. Ghent University. Belgium.

Vasarevicius, S. & Batavicius, T. (2013). Experimental Tests on the Influence of Waste Covering Layer on Odour Reduction. *Ecological Chemistry and Engineering*, 20(3), 543-554.

Van Broeck, G. (2011) Rapportage over de in MKROS geregistreerde meldingen van milieuhinder door Vlaamse gemeenten in de periode 1 januari 2006 tot 31 december 2010 (pp 1-39).

Vlaamse overheid & Leefmilieu Natuur en Energie (2012). 29 JUNI 2012. - Omzendbrief LNE 2012/1. - Milderende maatregelen voor geuremissies die afkomstig zijn van bestaande varkens- en pluimveestallen in Vlaanderen. In BELGISCH STAATSBLAD - 16.08.2012 - MONITEUR BELGE (pp. 48363-48371).

Vlaamse overheid, Departement Leefmilieu, Natuur en Energie (2013) Uitvoeren van een uitgebreide schriftelijke enquête en een beperkte CAWI-enquête ter bepaling van het percentage gehinderden door geur, geluid en licht in Vlaanderen – SLO-3, 254 p.

Vlaamse overheid & Leefmilieu Natuur en Energie (2016). Lijst met geactualiseerde emissiefactoren voor ammoniak, geur en fijn stof. In Bijlage richtlijnenboek landbouwdieren (pp. 1-29).

Walgraeve, C., Bruneel, J., Van Huffel, K., Demeestere, K., Vincze, L., De Meulenaer, B. et al. (2015). Sorption behaviour of targeted volatile organic compounds on airborne particulate matter using selected ion flow tube mass spectrometry. *Biosystems Engineering*, 131, 84-94.

Walgraeve, C., Van Huffel, K., Bruneel, J., & Van Langenhove, H. (2015). Evaluation of the performance of field olfactometers by selected ion flow tube mass spectrometry. *Biosystems Engineering*, 137, 84-94.

Wihnen, H. L. J. M. 1986. Guideline for Olfactometric Measurements in The Netherlands Comparison with Western European Guidelines. pp. 69-77. In: *Odour Prevention and Control of Organic Sludge and Livestock Farming*.

Willems E, Monseré T & Dierckx J (2016) Geactualiseerd richtlijnenboek milieueffectenrapportage "Basisrichtlijnen per activiteitengroep: Landbouwdieren", 151p.

Wing, S., Horton, R. A., Marshall, S. W., Thu, K., Taiik, M., Schinasi, L. et al. (2008). Air pollution and odor in communities near industrial swine operations. *Environmental Health Perspectives*, 116, 1362-1368.

Wzorek,B., P.Mochalski, I.Sliwka, and A.Amann. (2010. Application of GC-MS with a SPME and thermal desorption technique for determination of dimethylamine and trimethylamine in gaseous samples for medical diagnostic purposes. *Journal of breath research* 4:026002.

Yu, Z., Guo, H., & Lague, C. (2010). Livestock Odor Dispersion Modeling: A Review. *Transactions of the Asabe*, 53, 1231-1244.

Zarra, T., Naddeo, V., Belgiorno, V., Reiser, M., & Kranert, M. (2008). Odour monitoring of small wastewater treatment plant located in sensitive environment. *Water Science and Technology*, 58, 89-94.

Zarra, T., Naddeo, V., & Belgiorno, V. (2009). A Novel Tool for Estimating the Odour Emissions of Composting Plants in Air Pollution Management. *Global Nest Journal*, 11(4), 477-486.

Zernecke, R., Frank, T., Haegler, K., Albrecht, J., Brückmann, H., & Wiesmann, M. (2011). Correlation analyses of detection thresholds of four different odorants. *Rhinology*, 49(3), 331-336.

Zimmerman, B. (2015). Uncertainty of odour emission measurements - Study on an active area source. *Gefahrstoffe Reinhaltung der Luft*, 75, 412-416.

Other sources:

www.olores.org (2015a). A new standard to measure odours in Europe, the new EN 16841.

www.olores.org (2015b). Is this the end of n-butanol for olfactometry?

Statistics Belgium (data 2016)

http://statbel.fgov.be/nl/statistieken/cijfers/economie/landbouw/

VLAM, Vleesbarometer 2017, productiecijfers varkensvlees in België van 2016

Statistical office of the European Union (data 2016)

http://ec.europa.eu/eurostat/web/agriculture/overview

http://ec.europa.eu/eurostat/web/agriculture/data/main-tables

https://ec.europa.eu/agriculture/sites/agriculture/files/market-observatory/meat/pigmeat/doc/dashboard-pig_en.pdf

https://www.lne.be/milieuvergunningendecreet-vlarem-i-ii-en-iii

http://www.milieurapport.be/nl/publicaties/mira-onderzoeksrapporten/

http://www.compendiumvoordeleefomgeving.nl/indicatoren/nl0291-Geurhinder%3A-bronnen-en-beleid.html?i=13-45

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PUBLICATIONS

INTERNATIONAL JOURNAL WITH PEER-REVIEW

Hove, N. C. Y., Van Langenhove, H., Van Weyenberg, S., & Demeyer, P. (2016). Comparative odour measurements according to EN 13725 using pig house odour and n-butanol reference gas, *Biosystems Engineering*, 143, 119-127.

Hove, N. C. Y., Demeyer, P., Van der Heyden, C., Van Weyenberg, S., & Van Langenhove, H., (2017). Improving the repeatability of dynamic olfactometry according to EN 13725: A case study for pig odour, *Biosystems Engineering*, 161, 70-79.

CONFERENCE PROCEEDINGS

Hove, N., Van Langenhove, H., & Demeyer, P. (2012). Development of an olfactometric measuring facility according to CEN EN 13725 and to generate up to date odour concentrations from animal houses in Flanders. *Chemical Engineering Transactions*, 30, 97-102.

NATIONAL JOURNALS

Brusselman, E., Van Ransbeeck, N., Ulens, T., Hove, N., Demeyer, P. (2012) Luchtemissies uit Vlaamse stallen: Onderzoek ter ondersteuning van de sector en de regelgever. *MilieuTechnologie*, Vol. 9, 10.2012, blz. 2-7.

Duquenne, B., Demeyer, P., Hove, N., Herman, L. (2012) Neuzen in dezelfde richting: het nieuwe geurlab van ILVO is er! *Science & Technology Watch*, Vol. 92, 05.07.2012.

Degroote, T., Hove, N., Bossin, S. (2012) Geurhinder in kaart brengen. *Management & Techniek*, Vol. 8, 20.04.2012, blz. 6-7.

SCIENTIFIC ACTIVITIES

ORAL PRESENTATION - INTERNATIONAL CONFERENCE

September 24, 2012	Development of an olfactometric measuring facility according to CEN EN 13725 and to generate up to date odour concentrations from animal houses in Flanders. *International Conference on Environmental Odour Monitoring and Control (Nose 2012), Palermo, Sicily*

ORAL PRESENTATIONS - NATIONAL MEETINGS

May 24, 2013	The application of CEN EN 13725 for the measurement of odours from intensive livestock *ICT-Agri Air Cleaning Project, Merelbeke, Belgium*
January 10, 2013	Olfactometrie *Training day "Geur en geurhinder in de veehouderij", organised by Departement Leefmilieu, Natuur en Energie, Brussels, Belgium*
September 14, 2012	De sensorische meetmethode, olfactometrie *3 days' course on odour, odour nuisance and odour reducing techniques, organised by Inagro, Innovatiesteunpunt, ILVO and Ghent University, Merelbeke, Belgium*
June 19 - 26, 2012	Ervaringen met het Geurlabo op ILVO *The official opening of the odour laboratory at ILVO, Merelbeke, Belgium and the Vemis board meeting, Merelbeke, Belgium*

MSC THESIS

Hove, N. (2010). Variabiliteit in de nutriëntenopname door zoetwater macrofyten: een flume ^{15}N-labeling studie.

promotor: dr. ir. Natacha Brion; assistant: dr. Véronique Woule Ebongue

BSC THESIS

Hove, N. (2008). Carbon sources supporting epibenthic fauna in mangrove systems of the Betsiboka estuary (Madagascar): Importance of riverine inputs vs. local production.

promotor: prof. dr. Willy Baeyens, supervisor: dr. Perrine Mangion

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